

HOW TO

ANALYZE CLAY

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HOW TO  
ANALYZE CLAY  
PRACTICAL METHODS  
FOR  
PRACTICAL MEN.

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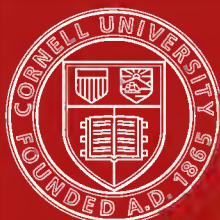
## INTRODUCTION.

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The purpose of this little work is to describe the analysis of clay so that a clayworker may make his own analyses with sufficient accuracy for all the practical purposes of every day work.

The methods which an experienced chemist uses with almost absolute accuracy generally gives very poor results in the hands of a beginner, with whom a far less accurate method would give better results; therefore, practical utility and chemical accuracy have been combined as far as possible.

In cases where the determination may be improved upon with greater experience, a second and more accurate method is also given.



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## HOW TO ANALYZE CLAY.



Our intention in this article is to tell the brickmaker in ordinary everyday English how to make a clay analysis that will be moderately accurate, and how to use it.

Chemists will probably laugh at the idea of anybody not specially trained, making such a difficult analysis as that of clay, nevertheless it is possible for an intelligent man to learn the routine of one single analysis, and be able to do it with sufficient accuracy to obtain practically useful results. Then it must be borne in mind that when a technical worker in clay has mastered even the simplest chemistry of his craft, his technical knowledge will lead him to make investigations which it would never occur to a chemist to make, and it is just this kind of research that is most needed.

There may also be some that will read this, who, having neither time nor inclination to make their own analysis, will realize that chemistry can help them if they will obey the laws of science, and if these laws are expounded to them in a way that they can understand.

For many years practical clayworkers have looked askance at the very mention of the word chemistry and with the best of reasons. Nowhere is the adage that a little knowledge is a dangerous thing better exemplified than in the application of chemistry to the arts; and up to a few years ago our knowledge of the chemistry of ceramics was in a very elementary state. Now, however, thanks to the magnificent example set us by both the German scientists and manufacturers, we are beginning to move in the right direction. Ceramic chemistry

has now been developed to such an extent that if the clay-worker will only listen to advice from the ceramic chemists, he will realize that it means a saving of actual dollars and cents. Until the hard times in 1893, brickmakers were in sufficiently easy circumstances to decline to trouble themselves with chemistry, but now, when a bare living is all that can be hoped for, a stoppage of a previous waste of from 2 to 7% will often mean easy circumstances. And that is just what chemistry has done in other cases and can do in this. One does not have to seek far for instances where chemistry has stepped in and utilized waste products or otherwise helped a business in such a way as to create fortunes.

Look at the Armour Packing Company, the Carnegie Steel Works, the United Alkali Company, etc. All these concerns could not pay such dividends but for the help they have received from science.

Now let us see just where the ceramic industry can be benefited by chemistry, and before going any further, perhaps it would be as well to state where to get our information. There is one solitary book that is written by an American, namely Langenbeck's "Chemistry of Pottery," and that, dealing as it does almost exclusively with enamels and glazes, does not quite meet our wants. For the main part of our information we have to rely almost entirely on the German publications with a few in French on the finer china ware. These foreign works are gradually translated and re-edited for the American public, in which work "Brick" is taking a leading part, but as yet they are not in a very accessible condition. For those who cannot study the foreign works, some information is to be gained from isolated magazine articles and portions of text books.

As most of our readers will be especially interested in the discussion of the chemistry of clay as applied to brick and tile making, we shall confine ourselves in the present article to that side of the question.

When a man starts out to make an analysis of a brick clay, what he wants to know finally is, what his clay is good for, what it will make, or, if he wants to make one certain thing of it, how to treat that clay in order to attain his end with the least possible outlay and waste. He does not care to know the exact proportion in which the different elements are present, because even to the cleverest ceramic expert the percent-



age of silica, alumina, iron, lime, water, etc., does not give any reliable data, but he does want to know whether the clay will make a good paving brick, or a good, rough building brick, and he does want to know what proportion of sand to add to make it a good fire brick.

To be able to do this, it is true, he must know the proportion of the elements, but that is only one step, and unless the others are taken the first step is useless.

To obtain such data as the color of the burned brick, it is best to burn a small piece of clay, because although we can make a pretty good guess from the chemical analysis, our information is not yet sufficiently accurate and varied to give us absolute results. The first step then is to have a small brick burned.

The next step is to mechanically divide up the clay into the various degrees of fineness; this can be done either by sifting or slumming. A large amount of useful information may be gained by this method, of which more hereafter, but by far the most valuable is the chemical analysis, so we will deal with this first. As stated above our final object is to find out what the clay can be used for, and to do this we have to first find out what the clay is composed of and then turn to records of previous analysis and see what results such component parts will be likely to produce. When we speak of the composition of the clay we do not mean the proportion of the elements present, but the proportion of real clay, sand, and undecomposed rock. Each of these bodies behave in a certain way when burned and if we cannot obtain data concerning other analysis, we can always get a good idea of the behavior of the clay from the average of its composition, which of course we could never do from the mere proportion of its elements.

Now to state in a few words how the proportion of minerals is found before going into details. When a clay is treated with warm concentrated sulphuric acid for 12 hours all the real clay substance is dissolved, and the sand and undecomposed rock is left. The two are separated and the undissolved portion is subjected to chemical analysis. We know that the undecomposed rock is feldspath and that there is in feldspath a certain definite proportion between the alumina and silica, so we take out enough silica to combine with the alumina, reporting all the rest of the silica as free sand.

## ANALYSIS OF CLAY.

	Total Constit- uents.	Insoluble Residue.	Actual Clay Substance.
Silica .....	61.30	35.94	25.36
Alumina .....	18.87	2.50	23.03
Iron Oxide .....	6.66		
Lime .....	0.85		0.85
Magnesia .....	1.20	0.20	1.00
Alkalies .....	3.30	1.23	2.07
Water .....	7.82		7.82
	<hr/>	<hr/>	<hr/>
	100.00	39.32	60.13

The proportion between alumina and silica in feldspath is 3.51, so if we multiply the 2.5 of alumina by 3.51 we get 8.77 as the silica in the feldspath; this subtracted from 35.94 total silica, leaves 27.165 as quartz or sand. The rest of the undissolved portion of the clay was feldspath, so we have as the composition of the feldspath:

Silica .....	8.77%
Alumina and Iron Oxide.....	2.2%,
Magnesia .....	.20
Alkalies .....	1.23
	<hr/>
Total .....	12.70

Our quartz or sand we find to be 27.165%, therefore our actual clay substance is  $100 - (27.165 + 12.705) = 60.13\%$ .

We thus have the following results:

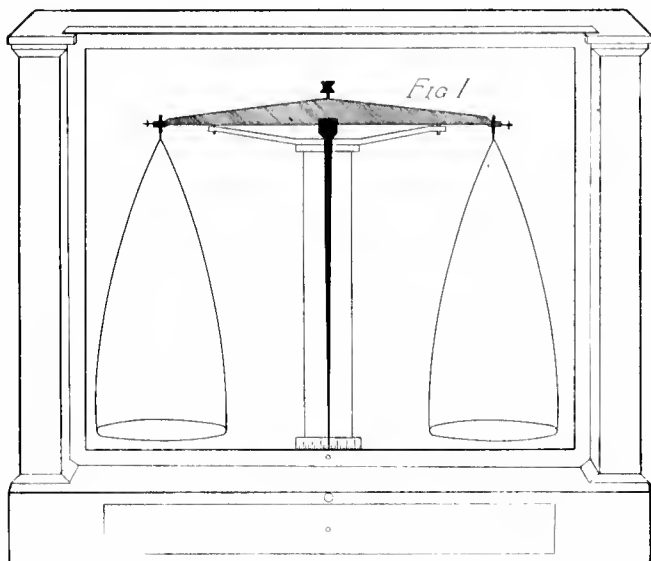
Sand .....	27.165%
Feldspath and Rock.....	12.705%
Clay .....	60.13%

To find the composition of that portion which dissolved in the sulphuric acid (the actual clay) we may analyze it or analyze the whole clay and then subtract the analysis of the undissolved portion.

Thus with two chemical analyses and a little calculation we know just what our clay is, and once the analysis is obtained it is easy to turn to a table of figures, which will be given later, to find the properties of the clay.

Now a few words as to the apparatus to be used. The outfit which will be described is not such as a consulting chemist

would think of getting, for a large amount of convenience and some accuracy is sacrificed to price and room, but what we want is something that will enable us to obtain useful results at the least expense. A man may purchase the balance described below, and after working for some time find that he can do enough work to make it worth while to get a more expensive one, but he will never regret having the old balance, as it will always be extremely useful. As to the rest of the



apparatus, the only difference between it and a high-priced outfit will be a question of quantity, not quality, so a man can easily add to the original stock should he go on to more complicated and difficult work. The prices quoted are the lowest likely to be given by any supply house, when ordering in this quantity.

The cheapest balance which will do the work with moderate efficiency is one of the good pulp or ore balances, preferably one made by Becker or Troemner, and when loaded to its full capacity should easily show the addition of one thou-

sandth part of a gramme. It should be fitted with movable pans, an attachment for resting the beam when not in use, and above all should have a glass case to keep off the draughts. (Fig. 1.) Such a one as that can be purchased for about \$17.

The weights should be second quality analytical, from 20 grams down to one milligramme (1-1000th of a gramme). all weights under one gr. being of aluminium. These will cost about \$2.60.

The platinum crucible, which is absolutely indispensable, is very expensive, but if the platinum is not spoiled by alloying with some other metal, which will only happen by the grossest carelessness or ignorance, it is always worth its weight for the metal alone, which is nearly as valuable as gold. It should be of the usual form, of 30 cubic centimeters capacity, weighing 30 grammes. This will cost \$15.

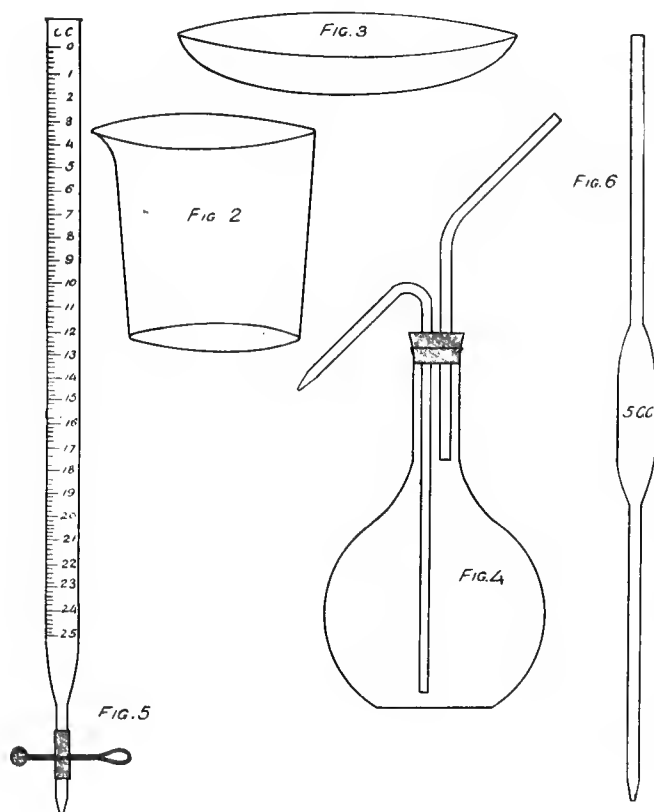
The question as to what burner is to be used presents some difficulty. In places where natural gas is laid on, and there is a good pressure, a large Bunsen burner is sufficient to fuse the clay in the crucible (with sodium carbonate), but where there is indifferent pressure this will not work and a blast lamp has to be used with the gas. Then again there will be a large number of places where there is no chance of getting gas at all, and for such the Hoskins gasoline blast lamp is probably the most serviceable; this costs about \$11.50, but it is such a generally useful article that it is well worth it.

These are the principal items of expense, the rest are small and will be described briefly. Of beakers, which are thin glass vessels for the purpose of boiling liquids in (Fig. 2), one should have a set of four, from 5 to 20 oz. capacity, and these should be of the form called Griffin's, which have a lip for facilitating pouring. One porcelain evaporating dish will be sufficient, barring breakage, of flat form, glazed inside, about  $4\frac{1}{2}$  in. diameter and 8 oz. capacity (Fig. 3). There should be three glass funnels, two of 2 in. diameter and one of 3 in.

The wash bottle may be made from a twelve oz. flask with rubber stopper and bent glass tubes, or may be bought ready fitted up (Fig. 4). The burette (Fig. 5), pipette (Fig. 6), and measured flask should be respectively of 25 cubic centimeters (divided in  $1/10$  c. c.), 15 c. c., and 500 c. c. capacity. The measured flask should be of the same shape as the wash bottle flask, with the graduation on the neck. One wedge-

wood pestle and mortar,  $4\frac{1}{2}$  in. diameter, will be sufficient for all ordinary purposes, and costs but fifty cents.

One iron stand or support with three movable rings, a pair of steel forceps about 6 in. long for taking hold of hot things,



and two packets of Munktell's No. 2 Swedish Filter paper 7 and 9 centimeter diameter, respectively, complete the list of apparatus. There will be several other things needed, but they can be made with a little ingenuity from ordinary

kitchen utensils. Some bottles will be needed, but as those chemicals which are in a liquid condition will be bought in suitable bottles, and for the half dozen solutions that have to be made up, ordinary medicine bottles will be good enough, it will not be necessary to include those in the list. The chemicals will be enumerated later on, but they should not cost more than \$3, so if we allow a margin of \$1.50 for various odds and ends, the total will be \$44, or \$55 if the gasoline lamp is used.

List of apparatus required:

Balance .....	\$17.00
Weights .....	2.60
Platinum crucible .....	15.00
Bunsen burner .....	.50
Beakers .....	.60
Porcelain evaporating dish.....	.25
Forceps .....	.25
Funnels .....	.30
Filter paper .....	.30
Wash bottle .....	.25
Burette .....	.70
Pipette .....	.20
500 c. c. flask.....	.45
Ring and stand .....	.75
Mortar .....	.50
Chemicals .....	3.00
Sundries .....	1.50
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Total .....	\$44.05

Before thinking of making any kind of an analysis one has to take particular care to have an average sample, and an average sample is a quantity of the substance to be analyzed, which represents proportionately all the various ingredients of the mass present at the time of sampling.

To sample a quantity of clay various methods may be employed. A small quantity may be taken from each stratum in the clay pit and these mixed together when dry, or if the clay presents a uniform appearance, small quantities may be dug out in different places and mixed.

If the clay has been dug up some time and is weathering in the yard, small quantities may be taken from different parts

of the heap; care being taken, however, not to have all the sample from the finer lumps or dust, or all of it from the large lumps, but equally from both.

The sample, weighing between 5 and 10 lbs., is now placed where it may be air dried until it will crumble into small pieces. It should then be ground or crushed until there are no lumps larger than a bean, and the whole mixed thoroughly together several times, and then spread out flat on a level surface. From this small quantities should be taken on the broad end of knife, from all over the pile, until about  $\frac{1}{4}$  lb. is obtained; this should be mixed thoroughly in a mortar, all the small lumps being broken by the grinding, and then put in a small bottle, corked up, labelled, and saved; the rest may be thrown away as we have now a representative sample of our clay. From this, which we call the rough sample, about 5 gr. must be taken, and ground in the mortar until there is no longer any gritty feeling when a small pinch is rubbed between the finger and thumb. As it is rather hard to grind a large quantity at once in a mortar, small portions should be taken at a time, and then emptied out. This, when ground, is the fine sample, and should be bottled and labelled ready for use, as it is what we weigh out for our analysis.

It will perhaps be as well to warn the beginner against leaving things around the shelves and tables on pieces of paper. One can never tell what impurities may fall in, and it is so easy to take a small bottle, paper box, or even tin can for clay, put the sample in, and last, but not least, label it.



## CHAPTER II.

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After sampling, probably the most important thing is to learn to weigh correctly on a fine balance. To begin with, the system of weights may bother some people, yet the metric or decimal system, which is always used in scientific and chemical work, is the simplest known. The measures of weight, length and volume used are so intimately connected that all may be explained at once.

A metre, the standard of length, is 3.2809 English feet, the centimetre is 1-100th of that, or 0.03937 inches, and the millimetre is 1-1000th of a metre and equals 0.03937 inches. In measures of volume we only use the cubic centimetre and the litre. The cubic centimetre or c. c., is the amount contained by a cubic vessel measuring one centimetre or 1-100th of a metre all ways. One thousand cubic centimetres equal one litre, and this is about equal to one quart. The unit of weight is the gramme, which is the weight of one c. c. of pure water at 0°. C. and normal pressure. In the laboratory we do not often use more than 200 grs., so do not use the decagramme, etc., but speak of so many grs. For smaller weights the gramme is divided into 1,000 parts, which are called milligrammes, so for weight we have practically two measures, the gramme and the milligramme. One gramme equals 15.432 grains Troy and one milligramme equals 0.015432 grains Troy. The set of weights referred to in the list of apparatus is brass for the grammes and aluminium for the milligrammes.

The following things should be carefully noted in using a chemical balance: Never weigh anything while it is hot, as the warm air arising causes the pan to rise also, and decreases the apparent weight. Never remove or replace anything unless the beam is at rest, because the knife edges are likely to be knocked out of place and blunted. Always before weighing see that the scale stands level and that the pans are equally balanced; if only a short time elapses between each weighing and no jar has been given to the balance, this precaution is not necessary.



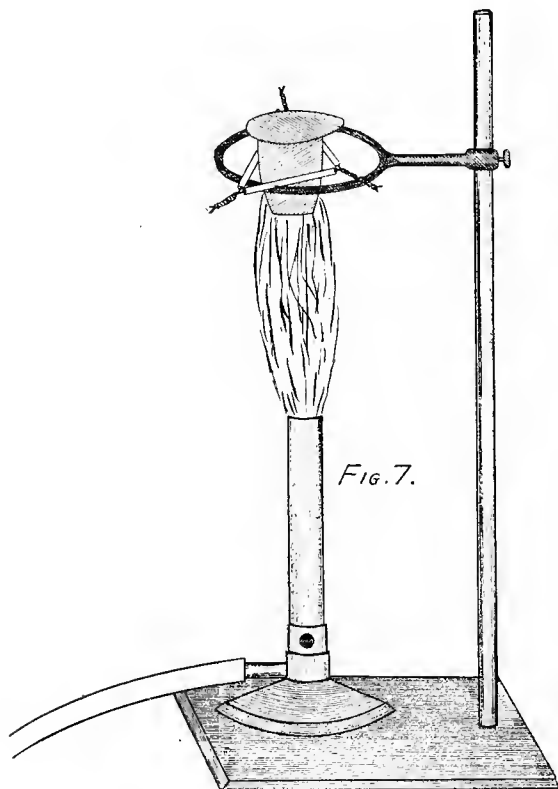
The actual operation is carried out thus: After seeing that the balance is level, and the pans clean and free from dust, put down the glass door and raise the beam by turning the little knob. If this is done very steadily, no swinging motion is imparted to the beam, but if a very slight jerk is given, not enough to jar the knife edges, the pointer will swing over a few divisions to one side of the center of the marked scale, and then back again a few divisions the other side. If the pointer swings the same distance each side of the center, then the balance is correct, if not the little screw at one end of the beam must be turned to add or take weight from that side. When the pans are evenly balanced, the object to be weighed must be put in one pan and then weights put in the other, beginning with the large ones and working down to the smaller, until the pointer once more marks the same distance each side of the center. Now the sum of the weights should be carefully noted, first by adding up from the vacant spaces in the box, and then checked by adding them as they are replaced.

Having now sampled the clay and learned how to weigh accurately, we have to take about 2 gr. of the prepared sample and place it in a small, smooth butter dish or watch crystal. This must now be put into an oven and heated at 100° C. (or 212° Fahr.) for one hour. An ordinary gas oven or even an ordinary saucepan or tin can placed over the Bunsen flame may be used if a thermometer is put in to insure the temperature not rising much above 100° C., but the simplest plan is to use an ordinary double boiler saucepan over a good fire and put the dish with the clay in the bottom of the inner pan, of course with water only in the outer one. This will keep the temperature about 99° C., which is hot enough to drive off the water that is not chemically combined. At the end of an hour this should be taken out and placed in the desiccator to cool.

The desiccator is a vessel intended for drying things in, and can be made from a small jar or butter crock with a piece of glass to cover the top, and some calcium chloride or concentrated sulphuric acid in the bottom to absorb moisture. A small support should be arranged to keep the articles that are being dried clear of the substance in the bottom.

We have now an average sample, finely ground, and thoroughly dried; enough might have been dried to suffice for the whole analysis, but as some days may intervene between the weighings, it is better to dry a fresh lot each time, as the clay

will absorb some moisture unless kept in a perfectly air-tight vessel. Of the two grammes in the dessicator we weigh out accurately one gr. and start on the determination of the total constituents. We start with this, instead of treating the clay



first with sulphuric acid, because the method of analysis is nearly the same in each case, so that one explanation will do for the two. The one gr. of clay is placed in the crucible and heated. The crucible may be weighed first and then the clay weighed into it, in which case the clay does not have to be re-

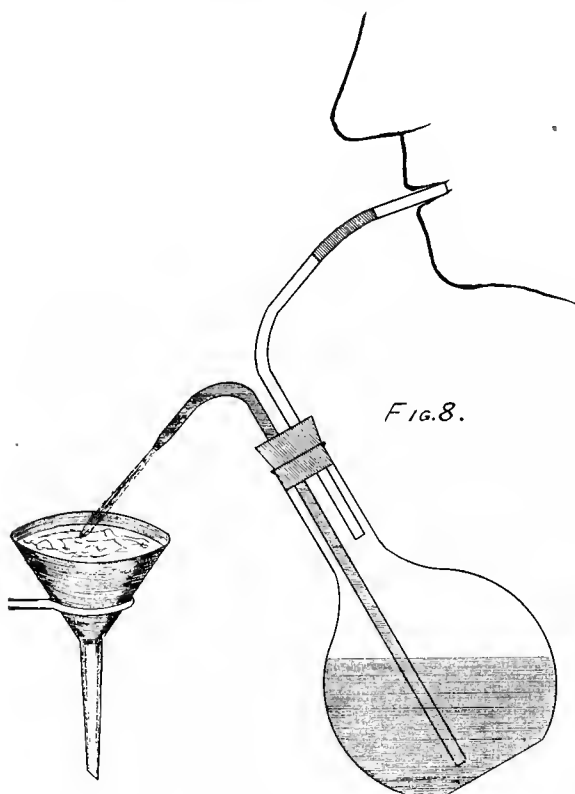
moved from the crucible, or the weighed clay may be brushed into the crucible without the weight of the latter being taken.

This is now placed on a pipestem covered wire triangle over the Bunsen flame (Fig. 7), at first with a small flame, and then a large one until a bright red heat is attained. This should be kept up for about 30 minutes, at the end of which time all the combined water and organic matter will have disappeared. The clay is then weighed and the loss is the organic matter and combined water. If one gr. is always taken for the analysis no figuring is required to find the percentage. One gr. is 1,000 millgrs., so if the loss were 25 millgrs. then the percentage would be 2.5

The clay that is left must be carefully mixed with 4 gr. of sodium carbonate and 1 gr. of potassium carbonate, both in a dried and finely powdered condition. This mixture is to be placed in the platinum crucible, the cover put on, and then heated with a low Bunsen flame for about 5 minutes. The heat must then be raised gradually until the mass is in a state of fusion, and should be kept so until no more bubbles appear to rise, and the whole is quite clear. This may be done either with the gasoline lamp or the Bunsen flame, as stated above. The gasoline lamp will accomplish it quite readily, but if the Bunsen is used some manœuvring may be necessary. The strongest possible flame should be used, not necessarily the biggest, but the hottest, and something should be put around the crucible to keep in the heat, preferably a section of clay pipe about three inches in diameter. If any furnace is convenient where the temperature is not too high the crucible may be placed in this with a piece of clay pipe around it.

When the mixture is quite clear and there are no more bubbles, it should be allowed to cool, and then put into a beaker and distilled water poured in to just cover the crucible lying on its side. This should be boiled for some time until the mass of the fusion is broken up and crumbles away, and then the crucible should be taken out, washed, and put away. Care must be taken to put all the washings in the beaker, so that nothing may be lost; a glass rod may be used to stir the fusion out of the crucible, but it should be washed into the beaker, also the tongs with which the crucible was lifted out. This washing should all be done with the wash bottle: if air be blown in at the upper glass tube a jet of water will be driven from the lower one, which can be directed to any point with great ease (Fig. 8).

When the fusion is completed and the mass separated from the crucible, an entire change has taken place in the constitution of the clay; the acids have combined with the sodium and the metals with the carbonic acid, so that we have sodium



silicate and aluminium carbonate, both of which are affected by acids, where before we had aluminium silicate, which is entirely unaffected by the ordinary acids.

If this be all treated with hydrochloric acid we obtain sodium chloride, aluminium chloride, silica, and carbon dioxide, the two former being soluble in water, silica insoluble, and carbon

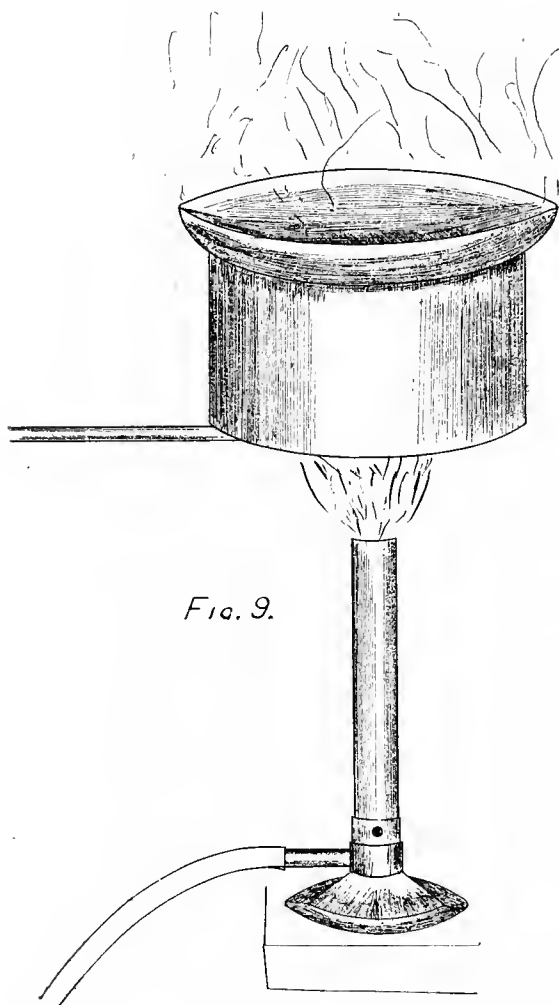
dioxide going off as a gas. Thus after the treatment everything is dissolved but the silica or sand. The method of treating the mixture with hydrochloric acid is somewhat long, but it must be followed exactly, as any deviation for the purpose of saving time is pretty sure to mean utter failure and a fresh start. The mass which is in the beaker should have about 4 oz. of water with it, if more has been used in washing the crucible, etc., it should be gently boiled until enough has evaporated, and then hydrochloric acid added little by little till there is no longer any effervescence, which is caused by the carbon dioxide gas escaping. When gas ceases to come off, all the carbonates have been converted into chlorides.

These must now be transferred to the evaporating dish and evaporated to dryness. If the volume is too great and fills the dish, a portion should be put in first, evaporated down, and then the rest put in. Great care must be taken to transfer everything from the beaker to the dish, as any loss means a loss of some of the ingredients of the clay.

Small transparent grains of silica may adhere to the sides of the beaker and they will be rather hard to see, but must all be taken off; they may be moved by a glass rod with a piece of rubber on the end, and then washed out with a stream of water.

When everything is transferred to the dish it should be placed on a water-bath, and heated to drive off all the water. A water bath is merely a metal vessel in which water can be boiled, and into which the evaporating dish will fit. (Fig. 9.) It is intended for keeping the temperature at about boiling point. Any iron or tin vessel on which the dish will rest so that part of it is inside and part outside will do. It should be kept about three-quarters full of boiling water. Any attempt at hastening matters by boiling over a flame will cause particles to sputter out with the boiling liquid, and spoil the analysis.

When a stage of complete dryness has been reached, about 2 oz. of pure hydrochloric acid should be poured on and again evaporated, care being taken that all the substance comes in contact with the acid. This must be once more evaporated to complete dryness, and acid added again and the evaporation repeated. When it is again dry about 4 oz. of a 10% sol. of hydrochloric acid should be poured on and all the solid matter stirred into it. When this has warmed for about 15 min-



utes on the water bath, it should be poured onto a filter paper and washed with warm water containing a little hydrochloric acid.

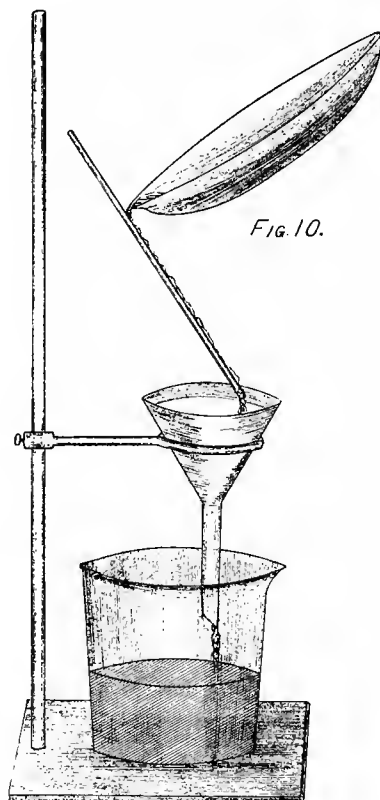
The operation of filtering is one which requires some explanation, but is almost the same in every case, so one explanation will do for all. The glass funnels mentioned in the list of apparatus have an angle of  $60^\circ$ , and if a circular filter paper is folded just in half, and then in half again, and opened out into a cone it should fit exactly into the funnel. In opening the folded paper, three thicknesses should be kept together to form one side, the other side being formed from one thickness of the paper. This will make a cone such that if filled with a mixture of a liquid and a solid the liquid or filtrate will pass through the pores of the paper, and the solid or precipitate will remain on the filter.

Some precautions are necessary in filtering for quantitative work, as this is a stage of analysis where beginners very often lose a large portion of their material, and, as stated above, any loss is fatal to accuracy. In the first place, the paper cone should fit exactly into the glass funnel, or at least the upper circumference should, because, the closer it fits, the quicker the operation, and less likelihood of loss. If, owing to a badly made funnel, the paper does not fit, it should be folded a little differently, not in quarters, so that the angle when opened out is more or less than  $60^\circ$ ; this must be found by experiment for each funnel. If the paper does not quite fit at the top, the pressure of the falling column of water, instead of being applied through the pores of the paper, is wasted on the space between the paper and the glass. Secondly, the funnel and filter paper should both be dry at first, and when a perfect fit is obtained, some water should be poured on from the wash bottle and allowed to run through, not poured off. The filter is then ready for use, and should be placed in the ring stand with a beaker underneath to catch the filtrate. (Fig. 10.)

The beaker should always be placed so that the end of the funnel just touches the side, in such a manner that the liquid will not drop, but slide down the sides of the beaker, in order to avoid splashing.

Never have the liquid in the funnel higher than within  $\frac{1}{4}$  in. of the top of the paper. In cases where the filtrate passes through very slowly, as in filtering from aluminium hydroxide, it is better to attach a piece of glass tubing about a foot long

to the end of the funnel by a rubber tube and allow the filtrate to pass through it. This gives a longer column of water, a greater pressure through the paper, and a correspondingly greater speed in filtering. Of course, this is absolutely useless unless



the filter paper fits the funnel accurately. In washing a precipitate on the filter, care should be taken that the stream of water from the wash bottle is not sufficiently violent to cause the particles to splash.



In the present case, namely, filtering from the precipitate of silica, enough of the mixture should be poured on to fill the filter to within  $\frac{1}{4}$  in. of the top of the paper, and the rest poured in as the liquid filters through.

None, either of the precipitate, which is silica, or the filtrate, which contains the alumina, iron oxide, magnesia, etc., may be lost, and in order to insure this, when apparently everything has been transferred from the dish to the filter, a small piece of filter paper, about  $\frac{1}{2}$  in. square, should be put in and moved over the whole surface with the glass rod, to loosen all the particles of silica; they can then be washed onto the filter with a jet of water. The piece of paper should be washed in with the rest and will be burned with the filter.

When everything has been transferred to the filter, the precipitate should be washed with about 2 oz. of warm water containing hydrochloric acid, the liquid being allowed to pass through into the beaker with the rest of the filtrate. This should be then set on one side, covered over, and labeled "Filtrate from Silica," and the precipitate on the paper should be thoroughly washed with about 6 oz. of the warm, acidulated water. We now have the silica, or silicon dioxide separate from the rest of the clay, but we have yet to prepare it for weighing.

The first step in this direction is to take the filter paper out of the funnel, place it in the drying oven, and dry it thoroughly, taking care that no impurities get in, or no loss occurs. While it is drying the platinum crucible should be cleaned out, dried and weighed.

On account of the value of platinum ware, some directions as to the use of the crucible may be in order here. None of the salts of silver, lead, or mercury should be put in the crucible, for if they be reduced to the metallic state they will invariably amalgamate with the platinum and spoil it. It will be better only to use the crucible when directed, in this scheme, and for any other work only if the user is quite sure that no harm can result.

Hydrochloric acid will not affect it, neither will nitric, or in fact any single acid, but if nitric and hydrochloric be mixed they will dissolve platinum, so great care should be taken that the two are never present together. In cleaning the crucible, if boiling water and the acids fail to remove the dirt, crystalline potassium di-sulphate should be put in, fused, and boiled

out with water. If this fails, a little fine sea sand, or the silica which is obtained from the clay analysis, may be used to rub away the dirt with.

When the crucible is washed and dried on a towel it should be heated to a dull red heat in the flame for a few minutes, placed in the dessicator to cool, and then weighed. We now have our silica and filter paper dried, and the platinum crucible weighed, and all that remains is to empty the silica into the crucible from the filter without spilling any, fold up the paper, put it in the crucible, and place the whole thing over a Bunsen flame.

When placing the silica in the crucible it is better to perform the operation over a piece of well-glazed paper, preferably black, so that if anything falls it may be brushed back into the crucible.

The precipitate has to be heated until the black carbon of the paper has all disappeared, leaving only the white silica and a little grey ash. It is better to tilt the crucible at an angle of about  $30^{\circ}$ , so that oxygen may enter to consume the carbon. When all the black has gone, the crucible may be placed in the dessicator to cool and then weighed. The difference in weight is the silica.

Thus we have determined the combined water and organic matter, and the total silica.



### CHAPTER III.

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On first reading this chapter a great mental confusion will probably be the only result. This need cause no discouragement, however, for even a practiced analytical chemist cannot read a new and complicated method with perfect understanding the first time. Even when the principle of the reactions is clear, the determination has still to be actually made before it is really understood. Therefore, let no man despair. First read it through, then follow the directions step by step, and if the reason for everything is not immediately clear, still the results will be attained, and after all that is the main thing.

In order to determine the silica we converted it into an insoluble form and filtered off all the soluble material, thus separating it from the other constituents of the clay. This method we pursue in nearly every case, the only difficulties being to find a sufficiently insoluble form of each constituent, and to arrange the order so that only one will be separated at a time.

Our next step is an illustration of the latter difficulty, for it is almost impossible to get out either iron or aluminium without the other, so we have to first separate the two from everything else, and then find a means of determining one in the presence of the other.

The aluminium and iron, be it remembered, along with the magnesium, calcium, etc., were all present in the filtrate from the silica in the form of soluble chlorides, with some considerable quantity of free hydrochloric acid present. If ammonium hydroxide be added to this, the soluble iron and aluminium chlorides are converted into the insoluble form of iron and aluminium hydroxides, when they may be separated from the other constituents by filtration; we then proceed to determine the iron by volumetric methods, that is, measuring it by volume instead of by weight.

The filtrate from the silica was allowed to pass through into a beaker, and such of the washings as were valuable enough were saved and added to the filtrate. The bulk of this may

vary according to the way in which the operator has handled the wash-bottle, but it does not much matter as long as the beaker, which should be a 12 or 15 oz., is not more than two-thirds full. If the bulk fills the largest beaker more than two-thirds it should be evaporated slowly, so that no particles jump out, until the volume is again within handy limits. Into this a small piece of red litmus paper should be dropped and ammonia water (ammonium hydroxide) added, little by little, the whole being constantly stirred with a glass rod, until the paper turns blue, and stays blue after stirring. This change will be preceded by a brown cloud appearing where the ammonia water first strikes the liquid, but the cloud disappears on stirring up the acid portions of the liquid. As soon as the litmus paper shows a distinct and permanent blue color, the acid has all been neutralized by the ammonia water, and the iron and aluminium chlorides converted to hydroxides..

Some excess of ammonia water, however, is pretty sure to have been added, and as aluminium hydroxide is slightly soluble in excess of ammonia, the beaker should be placed on a wire gauze and gently boiled to drive off the ammonia. When there is no longer any odor of ammonia above the liquid, the boiling may be stopped and the whole filtered while hot. This will proceed rather slowly, but can be hastened by adding a glass tube to the end of the funnel, as mentioned elsewhere.

When all the liquid has filtered through, about 6 oz. of boiling water is poured through into the rest of the filtrate, the whole set on one side, and labeled "Filtrate from iron and alumina."

A fresh beaker should now be placed under the funnel, and a warm mixture of half water and half hydrochloric acid poured onto the filter, until all the precipitate has dissolved and run through the paper. The latter should then be washed with about 2 oz. of warm water, the washings being allowed to run into the beaker with the dissolved precipitate.

The problem that now confronts us is to divide this solution, which contains all the iron and aluminium, into two exactly equal portions, and in one to determine the iron and aluminium together by weight as oxides, and in the other to determine the iron alone volumetrically. By subtracting the latter from the former, the amount of aluminium oxide is known.

If we had at our disposal all the various kinds of measuring

vessels, there would be no difficulty about the first part of this procedure, but as we only have one 500 c. c. flask some little invention will be required. A clean bottle, or glass vessel with a narrow neck, must be found, which will hold the liquid containing the iron and aluminium chlorides, and have some space left over, the less the better. Into this bottle the liquid is poured, and water added to fill it; the bottle is then emptied into some clean glass vessel, such as the 500 c. c. flask or a large beaker, the bottle once more filled to the mark with water, emptied into the larger vessel, and then the contents of this thoroughly stirred up. If now the bottle be thoroughly filled from the mixture, an exact half of the iron and aluminium is obtained. Great care must be exercised in this operation, both in stirring up the mixture thoroughly and in transferring from one vessel to the other without spilling any. It should also be borne in mind that any results obtained from either half must be multiplied by two to be correct.

One portion of the solution is to be saved and labeled "Half total iron and alumina for determination of iron," and the other portion emptied into a clean beaker ready for the determination of iron and aluminium.

It will be as well to state here that emptying any liquid from one beaker to another in quantitative work, the beaker from which the material is poured should always be rinsed out into the other one with a little water, to ensure nothing being lost.

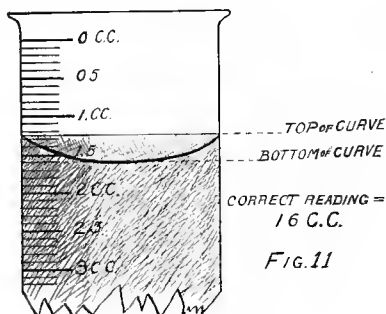
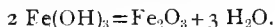
We now treat the iron and aluminium chlorides precisely as we did in the first place, putting in a piece of red litmus paper, neutralizing with ammonia water, boiling off the free ammonia, and filtering. In this case the filtrate need not be saved, as the amount of other constituents present should be too small (if the first precipitate of iron was properly washed), to be indicated on our balance. As soon as all the liquid has run through the filter paper, the precipitate should be washed with about 4 oz. of warm water, and after it has drained for a few minutes, placed in the oven to dry. When dry it should be put into the platinum crucible, which should be weighed first, ignited over the Bunsen flame until the black carbon has all disappeared, and placed in the dessicator to cool. When cool it should be weighed, the weight of the crucible subtracted, and the result multiplied by two, which will give the

amount of iron oxide ( $\text{Fe}_2\text{O}_3$ ) and aluminium oxide ( $\text{Al}_2\text{O}_3$ ) in one gramme of the clay.

Crucible and oxides .....	15.635 gr.
Crucible .....	15.370
	<hr/>
Oxides .....	0.265
	2
	<hr/>
	.530

Percentage of  $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = 53.0$

We stated above that the iron and aluminium were precipitated as hydroxides  $\text{Fe}(\text{OH})_3$  and  $\text{Al}(\text{OH})_3$ . When the hydroxides are heated, however, they give up water, and the oxides are left as follows:



We have now to determine the iron oxide and subtract that from the above result, which will give us the aluminium oxide. In order to do this, we have to depart from the gravimetric or weighing method, and turn to the volumetric or volume method.

Inasmuch as this is not a complete text book of chemistry, it would not do to spend too much time in explaining the principles of the work, yet a few words as to volumetric analysis will help to make things clearer.

If a match is lighted and placed in a bottle containing only a small amount of air the match will go out when all the oxygen of the air is consumed, and there will be wood (or carbon)

left, which has not been burned. On the other hand, if a lighted match is placed in a vessel containing plenty of air, the wood will be entirely consumed and some oxygen will be left.

Now let us assume that we have means of separating the oxygen from the other gases and measuring it, we know the proportion in which oxygen and carbon combine, therefore, without weighing the match we can tell the amount of carbon in it, and if we know the proportion of carbon in wood, we can also tell the weight of the match.

This principle we use to determine the amount of iron. We cannot easily separate the iron and weigh it by itself, so we find something which will react with it in the presence of aluminium, see how much it takes of this and then see how much iron there is. This method is based on the fact that chemical elements or groups of elements combine with one another only in certain definite proportions, which proportions we must learn in order to calculate our results.

Before arriving at any results, however, we have to learn something of the methods and appliances of the volumetric analysis. The term most frequently made use of, perhaps, is that of "standard solution." A standard volumetric solution is a solution of known strength of the reagent required. In using it, if a certain quantity is measured out, that quantity is known to contain a certain weight of the reagent, so that the weight is measured by the volume of the liquid, hence the name volumetric. For example, if a standard solution of iron chloride contains 10 gr. in the litre, each cubic centimetre contains one thousandth of that, or 0.01 gr. of iron chloride.

It is not always necessary to make a standard solution by weighing the reagent into a measured quantity of water, for often a solution of unknown strength may be standardized by comparison with some standard solution.

An indicator is some reagent which is added to the solution to show when the chemical action is gone far enough. The piece of litmus paper used for showing when enough ammonia had been added was an indicator.

The burette (Fig. 5) is used for dealing out the standard solution in any required quantity, within the capacity of the instrument, in such a way that the amount of liquid used can be measured at any time. In preparing the burette it should first be rinsed thoroughly with water, then again with a little

of the solution, in order to get rid of the water, and then filled up to the zero mark with the standard solution.

In such a narrow vessel the capillary attraction will cause the top of the liquid to assume the form of a curve, with the sides higher than the middle (Fig. 11), so in practical work all measurements are taken with this middle or bottom of the curve resting on the line or mark.

When the liquid is accurately adjusted to the zero mark, it can be run out into the beaker or receiving vessel by pressing together the clamps at the bottom, and when enough has been taken out the amount can be read off on the scale, the line on which the bottom of the curve rests being again taken. If more is now run out without refilling the burette, the first

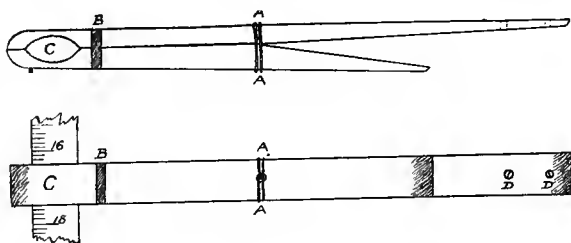


FIG. 12.

reading must be subtracted from the second to give the amount used.

The holder or support for the burette can easily be made with a pen-knife from two sticks of firewood and a rubber band (Fig. 12). The pieces of wood should be cut so that a point projects on each piece at A, where they may be tied together with string to form a hinge. B is a rubber band to clasp the two pieces tightly together at C over the burette. DD are holes for the screws which fasten the holder to the shelf. Needless to state that the holder must be put up in such a way that the burette is quite perpendicular (Fig. 13).

The pipette (Fig. 6) is an instrument for measuring a definite quantity of liquid, which quantity is always marked on the vessel. In using it the point should be inserted in the liquid to be taken, the mouth of the operator placed over the other



end, and the liquid raised by drawing in the breath, to a little above the mark on the neck of the instrument, the mouth quickly taken away and the first finger put in its place. It will be obvious why the liquid was raised to a point higher than the mark, for in replacing the mouth with the finger some is lost. The liquid can now be slowly lowered by slightly raising the finger, till the bottom of the curve rests on the mark (Fig. 14).

Before trying to measure any poisonous or objectionable

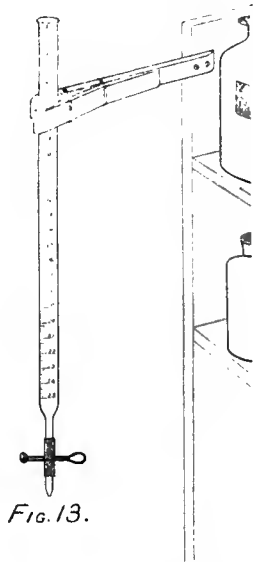


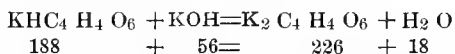
FIG. 13.

liquid with the pipette, the beginner had far better practice with plain water.

The determination of iron by the volumetric method is rather complicated, so, perhaps, it would be well to illustrate the principle by one of the simplest determinations. Let us suppose a case in which cream of tartar is mixed with other organic matter from which it cannot easily be separated. We know that no alkali or other acid is present, therefore any neutralization must be with the cream of tartar as the acid. We proceed to determine the amount of cream of tartar by

neutralizing it with a standard solution of caustic potash. This solution contains the molecular weight, or 56 grs. of caustic potash, in the litre, therefore, every cc contains 0.056 grs.

Five gr. of the cream of tartar mixture is weighed out, dissolved in a little water, a few drops of red litmus solution added, and the potash solution dropped in from the burette with continual stirring, until the litmus just changes to a permanent blue or purple color. The acid cream of tartar or potassium bi-tartrate has now been neutralized to potassium tartrate, according to this equation:



Therefore, 56 gr. of caustic potash combine exactly with 188 gr. of cream of tartar, or one gr. of potash with 3.357 gr. cream of tartar.

Let us suppose that we took 21 cc of potash solution. That equals  $21 \times 0.056$  gr. or 1.176 gr. of potash, and the cream of tartar present in the 5 gr. was  $3.357 \times 1.176$  or 3.9478 gr. Thus the percentage of real cream of tartar was 78.9.

The determination of iron, as stated above, is not so simple, yet follows on much the same lines. If sodium thiosulphate be added to ferric chloride ( $\text{FeCl}_3$ ) in solution, the iron gives up part of its chlorine, and is reduced to ferrous chloride ( $\text{FeCl}_2$ ), and the bright, yellow color is lost. This loss of color, however, is not sufficiently noticeable to be relied upon as an indicator for accurate results, so an excess of thiosulphate is always added, and then a standard solution of iodine added until all that part of the thiosulphate not acted on by chlorine has been acted on by iodine. When this has happened the solution will give a blue color with starch paste, owing to the presence of free iodine.

The results are arrived at in this way. The relative strength of the iodine solution and the thiosulphate solution is known, therefore the amount of the latter equal to the amount of iodine solution used, is subtracted from the total amount of thiosulphate used. The remainder is the amount required to reduce the ferric chloride present. The relation between the ferric chloride and the thiosulphate solutions has been determined by experiment, therefore the amount of iron present is known.

The standard solutions take some time to make, but once

made and the strength accurately determined, 10 or 12 determinations of iron can be run off in an hour.

The different solutions are prepared as follows:

#### I. FERRIC CHLORIDE.

Some clean, soft iron wire is obtained and 5.02 gr. accurately weighed out. This amount equals 5 gr. of pure iron, the rest being principally carbon. This wire is placed in an 8 or 10 oz. beaker, some concentrated hydrochloric acid added to dissolve it, and covered with a piece of sheet glass. When the iron is dissolved, with heat, if necessary, a few crystals of potassium chlorate are added to ensure the ferric chloride being obtained, and the solution boiled gently for some time to drive off the chlorine. The beaker must be covered all the time with a piece of glass or a watch crystal, and anything that has spattered onto the glass be washed off afterwards into the beaker. When all trace of chlorine gas has disappeared, and the solution cooled, the latter should be poured into the 500 cc flask, the beaker thoroughly washed out, the washings being poured into the flask, and the flask filled up to the mark with water.

This gives a solution of known strength, containing 10 gr. of pure iron in the litre.

or 28.9 gr. of ferric chloride,

or 14.28 gr. of iron oxide.

The sodium thiosulphate solution is standardized on this iron solution, therefore for the equivalent of every single cubic centimetre of ferric chloride used in the analysis there is 0.01428 gr. of iron oxide in our sample. When the iron solution has been made up to 500 cc it may be kept in an ordinary bottle and the flask washed out for the next operation.

#### II. SODIUM THIOSULPHATE.

Twelve grammes of the pure crystallized salt (commonly called hyposulphite of soda, or photographers' hypo) should be dissolved in water and the solution made up to 500 cc. This is not yet standard, its strength has to be determined by comparison with the ferric chloride.

#### III. CUPRIC SULPHATE.

Only a small quantity of this is required, about 100 cc and the strength is 1 in 100.

## IV. POTASSIUM SULPHOCYANATE.

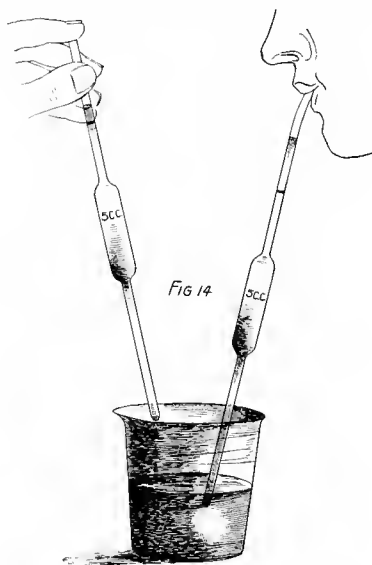
Also 1 in 100.

## V. IODINE SOLUTION.

Dissolve about 4 gr. of potassium iodide in water, add 2.5 or 3 gr. of pure iodine and dilute to 500 cc. The strength of this solution has to be ascertained by comparison with the thiosulphate.

## VI. A VERY THIN STARCH-PASTE.

The standardizing of the solutions is done as follows: Ten



cubic centimetres of the thiosulphate solution is measured off with the pipette into a beaker, a few drops of starch-paste added, and iodine solution run in slowly from the burette until a faint but permanent blue color appears.

Let us suppose that 13.5 cc of iodine solution was used, then 10 cc thiosulphate solution equals 13.5 cc iodine solution. This gives us the relation between the two solutions.

To compare the iron and thiosulphate, 10 cc of iron solution is measured into the beaker with a pipette, then 2 cc of con-

centrated hydrochloric acid, 100 or 150 cc of water, 3 cc of copper solution, and 1 cc of potassium sulphocyanate are added. The 3, 2 and 1 cc must be guessed at with the help of the 5 cc pipette, they are only approximate measurements. The burette is now washed out, rinsed and filled with thiosulphate solution. This is cautiously added to the mixture of iron chloride, etc., until the latter loses its color, and the amount of the solution added noted. The burette is emptied, washed and filled with iodine solution, some starch-paste added to the mixture in the beaker, and iodine run in until the blue color appears.

The amount of thiosulphate equivalent to the amount of iodine added, must be subtracted from the total of thiosulphate used; the remainder is the amount of thiosulphate solution equal to 10 cc of iron chloride solution.

Thus, if the total amount of thiosulphate were 12.5 cc and the iodine 3 cc, the amount to be subtracted would be 2.22 cc, leaving 10.25 cc, the proportion being 13.5:10::3:2.22. Therefore, in this supposed case, 10.28 cc of thiosulphate solution=10 cc of iron solution, or 1 cc = 0.97 cc.

The figures once obtained, our solutions are all standardized and ready for work, and we can now proceed to the actual determination.

The solution containing half the total iron in the clay was put in a bottle or beaker and saved. This is now taken, a few crystals of potassium chlorate added, the solution gently boiled, to drive off the chlorine, hydrochloric acid, water, copper solution, and sulphocyanate added, the same as before, thiosulphate run in till the color disappears, then starch and iodine till the appearance of a blue color. In fact, the operation should be conducted in precisely the same manner as in standardizing the solutions. When the equivalent of the iodine used has been subtracted, the real amount of thiosulphate and its equivalent of iron found, the determination is finished. To figure the results multiply the number of cubic centimetre of iron solution equivalent to the amount of thiosulphate used, by the factor 0.01428. This gives the amount of oxide of iron present in the sample. In our case this must be multiplied by two to give the amount present in one gramme of the clay.

We have previously determined the sum of the iron and aluminium oxides, therefore, if the iron oxide just found be subtracted, the aluminium oxide is also known.

## CHAPTER IV.

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When the iron and aluminium were precipitated for the first time, the filtrate was put on one side and labelled "Filtrate from iron hydroxide." This, as stated before, contains the calcium magnesium sodium and potassium in solution in the form of chlorides and hydroxides, and our next step is to isolate the calcium and magnesium. The question of determining the sodium and potassium will present some difficulty, but will be discussed later.

At this point especial attention must be given to the fact that we have several grammes of sodium and potassium chloride in solution besides the actual ingredients of the clay, these having been used in the fusion, therefore each precipitate must be very thoroughly washed, not only in order to get all of the filtrate but still more to free the precipitate from the foreign bodies mentioned. This is particularly to be noted in the case of the precipitation of iron and aluminium, where we dissolved and reprecipitated the hydroxides to free them from impurities, and also in the precipitation of calcium and magnesium.

In the case of the two latter, reprecipitation may be avoided if careful attention is paid to the washing, and the directions implicitly followed.

As before, we shall endeavor to convert the constituent to be determined into an insoluble form, in such a way that it is the only insoluble substance present. The form best suited to our purpose for calcium is that of calcium oxalate, which is practically the least soluble of the calcium salts, and on ignition is converted into either calcium carbonate or quick-lime.

### DETERMINATION OF CALCIUM.

The method is as follows:—The filtrate from the iron should be placed in a beaker large enough to contain twice its bulk, heated to boiling point, and about one gr. of crystals of ammonium chloride added, or the equivalent amount of ammonium chloride solution. Ammonium hydroxide must now be

added till a faint odor of ammonia is noticeable above the liquid after stirring, and then enough solution of ammonium oxalate to precipitate all the calcium and convert the magnesium into oxalate. The amount may be determined by adding the solution until no more precipitate occurs, and then as much again; or, if the oxalate solution be 10% (10 gr. salt in 100 c.c. water) 20 c.c. should be enough for any common clay.

The mixture should be thoroughly stirred up with a glass rod, the latter being left in the beaker, covered and allowed to stand perfectly undisturbed for some time, in order that the precipitate may all settle to the bottom. When this is done, a filter paper should be prepared, and the clear fluid filtered through into a clean beaker, care being taken that the precipitate is not stirred up. When no more liquid can be poured in without some of the precipitate going with it, the operation should be stopped, hot water (about 3 oz.) poured into the beaker containing the precipitate, and this stirred up again and allowed to settle as before.

The clear fluid should again be poured through the same filter paper into the rest of the filtrate, this time, however, to the last drop. When all the liquid is poured through, some more hot water should be stirred up with the precipitate and this poured slowly onto the filter paper, the object now being to transfer all the precipitate to the filter. When everything has been transferred except the few crystalline grains that adhere to the sides of the beaker, a small piece of filter paper may be dropped in and moved over the surface of the beaker with the glass rod, and then rinsed into the filter with the wash bottle.

Up to this point the filtrate should all be collected in the one beaker, which should be put on one side and labelled "Filtrate from calcium," and all subsequent washings thrown away. The calcium oxalate has now to be washed with another 6 oz. of hot water, after which it should be allowed to drain and put in the oven to dry.

In weighing our calcium we have the choice of several methods, but only the two most suitable will be discussed here. The calcium on the filter paper is in the form of calcium oxalate, which is a combination of the mineral base lime and the organic acid oxalic acid. Like most organic substances oxalic acid is destroyed when raised to a high temperature and converted chiefly into carbonic acid gas, so it

requires considerable care to burn the filter paper without the calcium carbonate giving up its carbon-di-oxide. But if the temperature is raised sufficiently, and the carbonic acid gas entirely driven off, caustic lime is the result, and our precipitate can easily be weighed as such. Therefore if the operator has a blast lamp or any other means of attaining a white heat in the platinum crucible, the second method is much simpler and quicker, if not, the first must be followed.

#### FIRST METHOD.

Take the clean platinum crucible, note the weight and place it over a shiny piece of paper, perfectly black. Then detach the filter paper containing the calcium oxalate without spilling anything, empty as much of the precipitate as possible into the crucible, and run a platinum wire or long steel hat pin through the empty filter paper, hold it over the crucible, set fire to it, and when burnt allow the ash to fall into the crucible.

This must be done where there is no draught, and if any of the precipitate or ash falls onto the paper, it should be brushed into the crucible with a small clean camel's hair brush. The filter paper is not reduced to an ash until it turns white or grey, and should not be dropped into the crucible in a half burned condition.

The crucible with the cover on is placed on a triangle and heated very gently with the bunsen flame for about ten minutes, and then the heat raised till the bottom of the crucible shows a faint red color, and kept thus for 15 minutes.

It is better at this stage for the operator to keep moving the flame back and forth with his hand in order that the heat may be equally distributed. At the end of 15 minutes the crucible may be cooled in the dessicator and weighed. If there has been any carelessness in heating the precipitate, and the carbon-di-oxide driven off, this may be detected by adding a few drops of water to the precipitate after it has been weighed, and testing the solution with a slip of turmeric paper.

If the paper does not turn brown, everything is right; if it does caustic lime is present and the paper must be rinsed off into the crucible, a small lump of pure ammonium carbonate added, and the precipitate evaporated to dryness by placing the crucible on an asbestos mat and applying a very gentle heat. The heat must not be sufficient to cause any sputtering



of the precipitate. When dry the latter should be heated on the clay triangle as before, only more carefully. After 15 minutes of a very faint red heat it may be cooled and weighed again. The ammonia carbonate is added for the purpose of supplying the lost carbonate acid gas, the ammonia and the unused ammonium carbonate being driven off at a very low temperature. Each gramme of calcium carbonate contains 0.56 gr. calcium oxide, so the result must be multiplied by 0.56 to give the calcium oxide.

#### SECOND METHOD.

The precipitate is placed in the weighed crucible and the filter paper burnt, precisely as in the first method. The crucible containing the precipitate and ash is placed on the support of the blast lamp and the temperature slowly raised to a very bright glowing heat. This should be kept up for about 10 or 15 minutes, then the flame turned off, and the crucible allowed to cool down to its ordinary appearance, then placed in the dessicator.

When quite cool it should be weighed, then heated once more for five minutes over the blast lamp, cooled and weighed again. If the two weights are the same, the determination is complete, but if the second is less than the first, the heating must be continued until no further loss in weight results. When the final weight is found the crucible must be subtracted, the difference being calcium oxide or quick lime. This amount must be multiplied by 100 to give the percentage.

The crucible should be wiped out with a clean dry cloth, and weighed, to see that it has not lost weight by the prolonged ignition; if it has, this amount must be added to the lime.

#### DETERMINATION OF MAGNESIUM.

We precipitate the magnesium as ammonium magnesium phosphate  $\text{NH}_4 \text{Mg PO}_4 + 6 \text{H}_2\text{O}$ . On heating this to  $100^\circ\text{C}$ . the  $6 \text{H}_2\text{O}$  is given off, and when the temperature is raised to a red heat, ammonia gas and water are given off, and magnesium pyrophosphate ( $\text{Mg}_2 \text{P}_2 \text{O}_7$ ) is left. It is in this form that we weigh it. Each gramme of this precipitate contains 0.36036 gr. of magnesium oxide or magnesia.

The determination is as follows:—The filtrate from the calcium determination is placed in a sufficiently large beaker,

some ammonia water added and then about 50 c.c. of a 10% solution of sodium ammonium phosphate. If this salt cannot be obtained sodium orthophosphate may be used, but must be mixed with some strong ammonia water.

The precipitation in the beaker should be carefully stirred in such a way that the stirring rod does not touch the sides of the beaker, as the precipitate has a tendency to crystallize wherever the rod has rubbed on the glass. Unless the amount of magnesium present is quite considerable there will be no precipitate at first, as it forms very slowly, so the beaker must be set on one side for some hours and then, if it is still quite clear and nothing has settled at the bottom, it may be assumed that there is no magnesium present.

If, however, there is any precipitate at all, either in suspension or settled at the bottom, the fluid must be filtered. As this is the last determination with this portion of clay, the filtrate need not be saved if it runs through quite clear.

When the precipitate has all been transferred to the filter paper, with the help of a piece of filter paper and the glass rod, and a specially prepared solution of ammonium hydroxide, it must be thoroughly washed with this latter solution.

The reason we wash with a solution of ammonium hydroxide instead of with water, is that the precipitate is slightly soluble in the latter, but not in the former. To prepare it, mix one part of ammonium hydroxide with three parts of distilled water. To see whether the precipitate has been thoroughly washed a few drops of the filtrate should be caught on the lid of the platinum crucible and heated in the flame. As long as any residue is left, the washing must be continued.

There is a possibility of an inexperienced operator finding some difficulty in handling the wash bottle filled with ammonia water, for if the mouth be suddenly withdrawn from it a stream of ammonia gas will rush back and cause considerable inconvenience. This is to be avoided by connecting a short length of rubber tubing with the mouthpiece, and compressing this so as to close it before withdrawing the mouth. Care must also be taken that the flask containing the distilled water is not confused with that containing the ammonia.

After the precipitate is washed completely it should be allowed to drain, then dried in the oven and transferred from the filter paper to the weighed platinum crucible, and the filter

paper burnt as in the determination of calcium. The crucible must be exposed to a gentle heat with the lid on for some time, and then heated over the blast lamp. If after heating for about 10 or 15 minutes the precipitate is dark colored it should be allowed to cool, a few drops of nitric acid added, warmed till dry and ignited again over the blast lamp. It may then be weighed and the result multiplied by 0.36936 to give the amount of magnesium oxide or magnesia.

Thus we have determined water and organic matter, silica, alumina, iron oxide, lime and magnesia. The elementary analysis is not yet complete unless we determine the sodium and potassium, but this is a question of considerable difficulty to the inexperienced chemist, owing to several causes. Neither of these metals form any compound which is very insoluble, so that they are hard to determine in any case. When added to this we consider that they are present in clay in very small portions, and also that we use both sodium and potassium salts for the fusion of the clay, it is evident that the question is much complicated.

Of course we cannot determine the alkalis in the fusion with the carbonates, so we have to find some other means of getting the clay in solution. The method generally used is to drive off the silica with hydrofluoric acid, when the alumina, iron, calcium and magnesium oxides may be precipitated, leaving the alkalis in solution. This, however, is tedious and takes nearly as long as the complete analysis, so, many chemists calculate the alkalis by difference, when they are not specially called for. This means that the percentages of all the other constituents are added together and subtracted from 100, the difference being assumed to be the alkalis. This is open to serious objections, as any unusual constituents which have not been determined and any errors in the work will affect the amount of alkalis found, but notwithstanding this it will probably be better for the beginner to be content with this method until he is more proficient in chemical manipulations. The correct determination, however, is performed as follows:

#### DETERMINATION OF ALKALIES.

Minute directions concerning the operation will not be given here, as the operator is supposed to have learnt how to

weigh, filter, etc., before attempting this determination so the following general directions will be sufficient:

First and foremost, nothing must be lost, as any loss at any stage of the procedure means an error in the result. When boiling or evaporating nothing must be allowed to jump over, so that it is better to boil very gently and to keep the vessel covered with a piece of glass, crucible cover, etc., which must be always rinsed off into the liquid afterwards. In filtering, care should be taken to leave none of the precipitate behind and to lose none of the filtrate (when it is needed) either by splashing, or taking out the glass rod and laying it down on a dirty table, or by not rinsing out the beakers, etc., etc.

The next point is to avoid adding any foreign substance to the material, as by leaving a beaker, or funnel containing a precipitate, uncovered, so that dust may fall in, etc.

It will also be advisable to make the determination right through to the end for the first time, notwithstanding such mistakes as a little loss or a little gain, so that a general idea may be had of the method and the first mistakes avoided the second time.

One gr. of the clay, prepared exactly as for the general analysis, is accurately weighed out and placed in the platinum crucible. It is then moistened with a few drops of water, about 1 c.c. of concentrated sulphuric acid added, and then from 5 to 10 c.c. of hydrofluoric acid. This acid must never be allowed to come in contact with any glass vessel, so the quantity will have to be guessed. The crucible is placed on the water bath and heated until the fumes of hydrofluoric acid and silicon fluoride have ceased to come off. If a home-made water bath is used, a piece of tin must be cut with a hole into which the platinum crucible will slip about half way down.

When no more fumes come off, the crucible should be placed over a bunsen flame on a piece of asbestos and heated at a high temperature till dense white fumes of sulphuric acid begin to come off. It should then be allowed to cool, washed into a large beaker with plenty of water, neutralized with ammonia water, of which a little excess is added, then some ammonium oxalate, and warmed gently for a time. Enough water should have been added to bring the volume up to about 400 c.c. after the addition of the reagents. This mixture when cool, is poured into the 500 c.c. flask, the beaker rinsed into the flask,

the latter filled exactly to the 500 c.c. mark, and then the whole allowed to stand until the precipitate has all settled to the bottom, leaving a perfectly clear liquid.

As much of this liquid as possible must be taken off either with a syphon or pipette and put into a clean, dry bottle and stoppered. This contains the magnesium, sodium and potassium as sulphates, and the precipitate contained the iron, aluminium and calcium, the silicon having all been driven off by the hydrofluoric acid. It is not necessary to obtain all of the filtrate, as we take 100 c.c., find the alkalies in that amount, and then multiply by 5. It is better, however, to have enough of the clear filtrate for 2 or 3 determinations, in case of mistakes.

One hundred c.c. of this is measured off by first rinsing out the clean burette with some of the liquid then filling it and running out exactly 25 c.c. four times over. Each 25 c.c. is run into the platinum crucible and evaporated to dryness over the water bath. When one lot is evaporated the next is run into the crucible and this evaporated, so that at last all the solid matter in 100 c.c. is collected in the platinum vessel.

This is then heated over the hunsen flame to drive off all the salts of ammonia. When no more white fumes come off, dissolve the residue in as little water as possible, rinse out into a small beaker, add a little ammonia water (about 15 c.c.) and some ammonium carbonate solution (20 c.c. of a 10 per cent sol.) and allow to stand for 24 hours. Then filter, wash with the ammonia and the ammonium carbonate solution, and evaporate the filtrate and washings once more in the platinum crucible. This takes out the magnesium, and as it has already been determined, the precipitate need not be weighed. Needless to state that the washing should be performed as carefully as possible, as the less liquid used, the less time will the evaporation take. Yet enough must be used to ensure all the alkalies being dissolved; 50 c.c. will probably be sufficient.

When the filtrate and washings are evaporated to dryness they must be dissolved again in a little water, a few drops of ammonia water added, filtered, washed with distilled water, and the filtrate and washings once more evaporated to dryness. But before the liquid is placed in the crucible this last time, it should be dried and weighed. When the evaporation is complete the crucible must be heated strongly over the blast lamp for a few minutes, cooled and then weighed. The differ-

ence in weight is the sodium and potassium sulphates found in 100 c.c. and when multiplied by five gives the amount in 1 gr. of clay.

The potassium has now to be determined separately and then subtracted from the sum of the two to give the sodium. In order to do this the residue in the crucible is dissolved in a little dilute hydrochloric acid and washed into a small porcelain crucible. Enough hydro-chlor-platinic acid (acid platinic chloride) is added to convert the alkalies into platinic chlorides and the whole evaporated nearly to dryness. The amount of hydro-chlor-platinic acid to be added must be left to the judgment of the operator, but about 10 c.c. of a 3 per cent solution will probably be enough for most cases.

When the residue is almost dry a mixture of 3 parts absolute alcohol and 1 part ether is added to dissolve the sodium platinic chloride, leaving the potassium salt undissolved. This is then carefully transferred to a filter paper which has been specially prepared, washed with the alcohol ether mixture, then dried for 30 minutes in the oven at 100°C. and weighed with the filter paper. To prepare the latter, fold it and dry for 30 minutes in the oven at 100°C. (in a clean place) and weigh when cool. If this weight be subtracted from that of the filter paper plus precipitate the weight of potassium platinic chloride in 100 c.c. sol. is known. Multiply this by 5 to find the amount in 1 gr. of clay, and then by 0.357 to convert it into potassium sulphate, subtract this from the sum of potassium and sodium sulphate, and the amount of sodium sulphate is found.

These must be reported, however, not as sulphates, but as oxides, so the potassium sulphate is multiplied by 0.54091 to give potassa or  $K_2O$ , and the sodium sulphate by 0.43694 to give the soda or  $Na_2O$ . These amounts are found in 1 gr. of the clay, therefore must be multiplied by 100 to give the percentage.

As the method of weighing the filter paper first and then weighing the paper and precipitate is liable to error owing to the fact that the paper may absorb moisture unless weighed immediately after drying, another method of weighing the potassium salt is given.

When the potassium platinic chloride has been washed with alcohol ether on the filter paper, which does not need to be dried and weighed, it should be dissolved in a little boiling

water and washed with the same, the filtrate and washings being run into a small porcelain crucible and evaporated nearly to dryness. To this should be added some small pieces of pure zinc, (about 5 gr. in all) and enough concentrated hydrochloric acid to dissolve it. The zinc should be tested first by dissolving a piece in the acid, and if it leaves no black residue it is pure enough.

The nascent (in the act of being generated) hydrogen generated by the zinc and acid will reduce the platinum salt so that the black metallic platinum is precipitated. When all action ceases, water should be added, the black residue poured onto a filter, washed with warm water, dried, and the paper and precipitate burned in the weighed porcelain crucible, and this weighed. The weight of metallic platinum multiplied by 2.461 gives the weight of potassium platonic chloride, which should be figured as before.



## CHAPTER V.

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A rough outline of the rational analysis has been given in an earlier chapter, but before going into detail on the subject it will be well to say a few more words as to its real value.

The composite mass known as common clay is generally made up of three separate minerals, real clay or kaolinite, feldspath, and quartz or sand. The relative proportion of each of these is a determining factor in the qualities of the burned product, such as the shrinkage, porosity, fire resistance, hardness, etc., therefore it is essential that we should know their relative proportions.

When we come to consider the components of these minerals and find that each contains very much the same elements, it becomes evident that an absolute separation would be very difficult, if not impossible. Kaolinite, or real clay, is hydrated aluminium silicate, having the formula (according to Seger),  $\text{Al}_2 \text{O}_3 + 2 \text{SiO}_2 + 2 \text{H}_2 \text{O}$ , or roughly:

Alumina $\text{Al}_2 \text{O}_3$ .....	40%
Silica $\text{SiO}_2$ .....	46%
Water $\text{H}_2 \text{O}$ .....	14%

This formula may be varied by the substitution of oxide of iron ( $\text{Fe}_2 \text{O}_3$ ) and occasionally oxide of manganese, for the oxide of alumina, and of the oxides of potassium ( $\text{K}_2 \text{O}$ ), sodium ( $\text{Na}_2 \text{O}$ ), magnesium ( $\text{MgO}$ ) and calcium ( $\text{CaO}$ ) for water.

Feldspath is much less simple, and has a more varying character. Seger, however, estimates the proportion of silica to alumina in this mineral as 3.51 to 1, and this is practically the only definite fact about its composition that we can make use of. The quartz or sand is extremely simple, being silica ( $\text{SiO}_2$ ) that is not combined with anything else.

The problem that now confronts us is to separate these three minerals, two of which are made up of the same constituents and the third being present in the other two in a combined form. Seger has devised the method of treating the whole



clay with concentrated sulphuric acid, which dissolves the kaolinite or clay proper, leaving behind the quartz and feldspath. This is weighed and analyzed, and separated into quartz and feldspath according to the assumed proportion of alumina and silica in the latter. No one will attempt to prove that this is in any sense an accurate method, but it is the best yet offered, and gives results which are far in advance of any other chemical analysis in practical value. Most clayworkers understand the relative importance of clay, spar and flint, and when the clay is separated into these its effect can be approximately determined.

The method is not accurate, in that the sulphuric acid leaves some of the kaolinite undissolved, and dissolves some of the feldspath, and also in that the constitution of both the kaolinite and feldspath is an assumption and not a proven fact. The actual method of determination is as follows:

#### RATIONAL ANALYSIS.

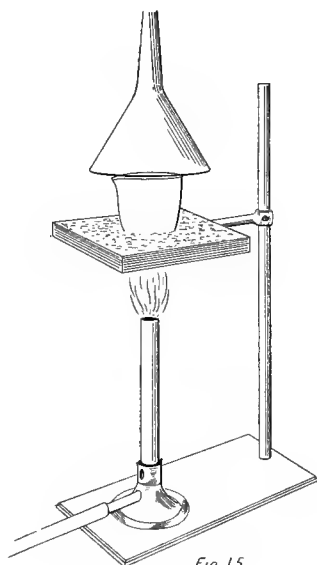
The clay is dried as before at 100° C., and 1 gr. placed in the platinum crucible. The whole operation may be performed in one of the fine Berlin porcelain crucibles if the operator finds too much difficulty in handling the amount of acid required in the small platinum vessel, but it is far better to use the latter if possible as the sulphuric acid has a slight action on the porcelain. Of course the best thing is to buy a larger platinum dish, but the expense will in many cases prohibit this.

When the weighed clay has been carefully transferred to the crucible it should be moistened with water, about 15 c. c. of concentrated sulphuric acid carefully added, and the crucible placed on the sand-bath. The temperature should be such that the acid does not fume or boil, and yet must be fairly high. When the temperature has been properly adjusted, some contrivance should be fixed a little above the crucible to keep out the dust, such as a large glass funnel inverted, and then it may be left alone for 10 or 12 hours, preferably the latter.

A sand-bath can be made of any lid of a tin can that has no solder in it, about 3 or 4 inches in diameter, or a piece of sheet iron can be bent up around the edges. This is filled with some fine sand, placed on the support over the bunsen flame, and the article to be heated put on it. (Fig. 14.) It ensures a

fairly equable temperature. Before using for the first time, however, the sand in the bath should be heated quite strongly so that if any water is imprisoned in the sand, it may burst out while there is nothing on the bath to spoil.

At the end of 12 hours the flame should be turned out and when the contents of the crucible are quite cool, the liquid decanted off into a glass beaker. This must be done so as not to shake up the undissolved portion. When nearly all the liquid



is poured out, water is carefully added to the mass in the crucible, stirred up, allowed to settle, and decanted into the beaker. This should be done twice to free from sulphuric acid. The addition of water in the first case must be made carefully as the strong acid becomes very hot when water is added, and may sputter out; the same is true when the second decantation is added to the first in the beaker. Some more water is now added to the liquid in the beaker and filtered,

care being taken that any solid matter present is caught on the paper. While this is filtering about 20 c. c. of a moderately dilute solution of caustic soda is poured into the crucible and heated almost to the boiling point for about five minutes. When the residue has settled down the clear liquid should be poured into the beaker, diluted with water and poured through the filter paper previously used. The operation should then be repeated with dilute hydrochloric acid on the residue in exactly the same way, then with caustic soda solution, and then again with hydrochloric acid. In each case the liquid should be decanted into the beaker, diluted with water and poured through the filter paper.

When the residue has been twice treated with hydrochloric acid, and twice with caustic soda it should all be transferred to the filter and washed with a very dilute solution of hydrochloric acid. None of the filtrate need be saved, either from the hydrochloric or sulphuric acids, or from the caustic soda. Especial care should be taken that none of the residue is left in the beaker or crucible. By the alternate treatment with soda and acid the limestone or calcium carbonate is all dissolved, and also such of the kaolinite as the sulphuric acid did not take out.

The residue on the filter paper is quartz and feldspath, and when washed should be dried, ignited, cooled and weighed. This weight subtracted from the 1 gr. of clay taken gives the amount of kaolinite plus limestone. The latter must be determined separately later on.

The analysis of the insoluble residue may be made in two ways, either by fusion with sodium and potassium carbonates, just as described for the total analysis, or by treating with hydrofluoric acid, in much the same way as in determining the alkalies. The former method is more suitable for beginners, but the latter is more accurate, as it determines the alkalies directly, and should be used when the operator is thoroughly proficient.

#### FIRST METHOD.

(Analysis of Residue, Insoluble in Sulphuric Acid.)

Three grammes of finely powdered sodium carbonate and one gr. of potassium carbonate are mixed together and the dried and weighed residue brushed into it and thoroughly mixed. This is fused in the platinum crucible, cooled and

treated with hydrochloric acid just as in the first analysis. When the silica is separated by filtration, washed and dried, it should be weighed. The filtrate is neutralized with ammonia water, filtered, washed, the filtrate put on one side for the determination of magnesia, and the precipitate of iron and alumina dissolved in a few drops of hydrochloric acid, reprecipitated with ammonia water, washed, dried and weighed. The details of the method have been given previously, and they should be followed accurately. It will not be necessary to separate the iron and aluminium as they are considered as one in figuring the feldspath.

The lime and magnesia are determined exactly as before, and to find the alkalies the silica, iron, alumina, lime and magnesia are added together and subtracted from the original weight of the residue. The amount of aluminium and iron found must be multiplied by 3.51 to give the amount of silica in the feldspath. This quantity of silica is subtracted from the total silica in the insoluble residue, and the rest reported as quartz or sand. To the silica and alumina of the feldspath must be added the lime, magnesia and alkalies.

If the analysis of the insoluble residue, for example, were:

Silica .....	35.94
Alumina and iron oxide .....	2.50
Magnesia .....	0.20
Lime .....	....
Alkalies .....	1.23
<hr/>	
Total .....	39.32

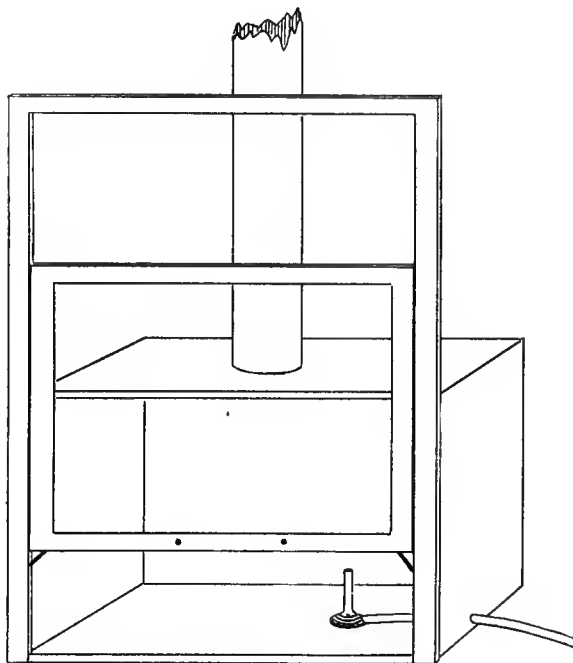
The 2.50 of alumina multiplied by 3.51 would give 8.77 silica, and the composition of the feldspath be as follows:

Silica .....	8.77
Alumina and iron oxide .....	2.50
Magnesia .....	.20
Alkalies .....	1.23
<hr/>	
Total .....	12.70

And the rational analysis of the clay would be reported as follows:

Kaolinite or real clay .....	60.63
Quartz .....	26.17
Feldspath .....	12.70

Before describing the second or hydrofluoric acid method, a few words may be in order as to the disposal of noxious fumes. Both in the determination of alkalies and in the present case, hydrofluoric acid has to be evaporated, and very

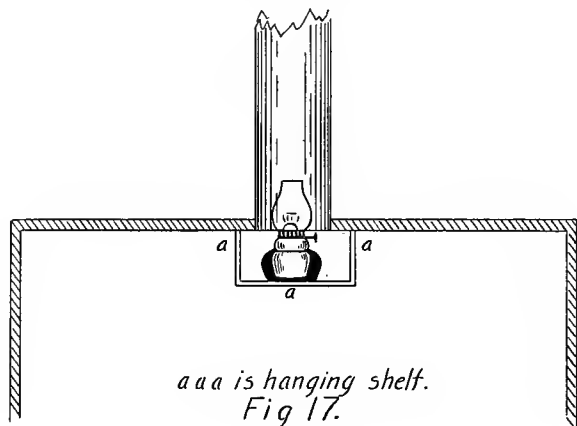


*Fig. 16*

frequently hydrochloric acid has to be driven off. If these fumes are allowed to escape into and fill the room, considerable inconvenience and some danger will inevitably result. Hydrochloric acid is not so bad, but hydrofluoric is really dangerous.

The contrivance for carrying off the fumes is called a hood or draught closet. A well made, close jointed box, about 2 ft. wide,  $1\frac{1}{2}$  ft. deep and  $1\frac{1}{2}$  ft. high, fitted with a sliding front (Fig. 16) will do very well. The front is better if it slides up

and down like a window, and should have a pane of glass let in for the operator to see what is going on inside without raising the front. A piece of stove pipe should be fastened over a hole in the top and connected with the chimney of the room. If after the connection is made, it is found that there is not sufficient draught to carry away the fumes, a small kerosene lamp should be placed in the lower part of the stove-pipe in order to create a draught (Fig. 17). All crevices and holes must be closed by pasting paper over, and the hood is made much more permanent by painting the inside and outside



of the stove-pipe with any oil paint or varnish. If gas is used a small hole may be bored in the side of the box for the rubber pipe to pass through.

#### SECOND METHOD.

##### (Analysis of Residue.)

The insoluble residue in the platinum crucible should be moistened with water, a few drops of sulphuric acid (about 1 c. c.) added and then about 10 c. c. of hydrofluoric acid. The precautions for the use of this acid mentioned in the determination of alkalis should be noted here. The crucible with contents is placed on the water bath and heated till no further odor of ammonia is noticeable above it.

When all the volatile acid has been driven off, the crucible is placed on the sand-bath or asbestos plate and heated until white fumes of sulphuric acid begin to come off. Then it is taken off, cooled, and the contents washed into a beaker. All the silica should have been driven off as hydro-fluo-silicic acid. If after dissolving the contents of the crucible in water there is any insoluble residue, this should be put back in the crucible, again treated with hydrofluoric and sulphuric acids, and washed back into the beaker. The solution in the latter contains the bases of the feldspath as sulphates, such as aluminium sulphate, magnesium sulphate, etc.

The solution should be neutralized with ammonia water, a little excess added, boiled gently for a few minutes, and filtered. The precipitate is iron oxide and alumina. It should be washed well, the filtrate and washings saved for the other determinations, the precipitate dissolved in hydrochloric acid, reprecipitated with ammonia, filtered, dried and weighed.

The lime may be determined in the first filtrate from the iron and alumina by either of the methods mentioned in Chapter IV., and the filtrate saved for the determination of alkalis and magnesia. These are found exactly as in the determination of alkalis mentioned in the latter part of Chapter IV., except that instead of diluting the filtrate to 500 c. c. and then taking 100 c. c., the whole of the filtrate from the lime is taken and evaporated to dryness. Also when the magnesium is separated with ammonium carbonate it should be washed, dried and weighed, either as magnesia ( $MgO$ ), after strong ignition over the blast lamp, or as magnesium carbonate after being heated to a gentle redness. The same precautions hold good as in the determination of lime. If the salt is determined as a carbonate, the amount is multiplied by 0.476 to give  $MgO$ . In calculating the results it should be borne in mind that the total does not have to be multiplied by five, as the whole of the filtrate was taken.

To express the results as feldspath and quartz exactly the same method of calculation is used as when the analysis is made with the sodium carbonate. Seger sometimes uses a way of calculating the feldspath without finding its constituents which will be found useful in many cases, but it is not so accurate as it does not allow for the other kinds of undecomposed rock.

The residue, insoluble in sulphuric acid, is treated with

hydrofluoric acid and the iron and alumina determined as already described, and then this amount of iron and alumina multiplied by 5.41 to give the feldspath. This is subtracted from the insoluble residue, the remainder being quartz. This is, of course, much quicker than the other methods, but is not so complete.

Before the analysis can be considered finished, carbonate of calcium or limestone should be determined. The determination of the total amount of calcium has been made and calculated as lime ( $\text{Ca O}$ ). When the clay was heated at a high temperature to drive off the water and organic matter, any limestone or calcium carbonate present would have been converted into lime, the carbonic acid gas being driven off. The latter was thus considered as organic matter, and on looking at the analysis one could not tell how much of the lime was combined with the silica and how much with carbonic acid. Inasmuch as the calcium carbonate has considerable influence on the clay, both while burning and afterwards, it should be determined separately. This may be done in several different ways, of which the following is probably the most suitable for our purpose:

#### DETERMINATION OF LIMESTONE.

About 20 c. c. of concentrated hydrochloric acid is thoroughly mixed with 50 c. c. of water and divided exactly into two equal parts. If this is found to be difficult two portions of exactly 25 c. c. each may be put into two separate beakers, the object being, of course, to get two perfectly equal quantities of about 25 c. c. A piece of hard white marble about the size of a small walnut should be selected, dusted and brushed to remove all loose particles, dried in the oven for an hour at  $100^{\circ}\text{C}$ ., cooled and weighed. If the weighed marble is now placed in one of the beakers, it will be dissolved by the hydrochloric acid until all the latter is used up. The liquid should be stirred every little while to allow all the acid to come in contact with the marble, the stirring rod being left in the beaker. When all action has ceased, that is when no more bubbles have come off for the last ten minutes, the marble is taken out, rinsed, dried in the oven for one hour at  $100^{\circ}\text{C}$ ., and weighed. This weight subtracted from the original weight gives the amount of marble that the hydrochloric acid in the beaker can dissolve.

While this is going on 5 gr. of the clay finely powdered and



dried at  $100^{\circ}\text{C}$ ., should be put in the other beaker and stirred up occasionally. Some time after all action has ceased a piece of marble, similar to the one used before, or the same piece, should be dried, weighed, placed in the beaker with the clay, dried and weighed when all action is over. The difference between the loss of weight of this piece of marble and the first equals the amount of calcium carbonate or limestone in the 5 gr. of clay taken.

### Synopses of Analyses.

#### General Analysis.

Take 1 gr. of clay, dried at  $100^{\circ}\text{C}$ ., heat in platinum crucible over blast-lamp, weigh.

Loss=Water and Organic Matter.

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Mix residue with 4 gr.  $\text{Na}_2\text{CO}_3$  + 1 gr.  $\text{K}_2\text{CO}_3$  and fuse.

Dissolve fusion in  $\text{H}_2\text{O}$  +  $\text{HCl}$ , evaporate solution on water bath, treat twice with  $\text{HCl}$ , dissolve in dilute  $\text{HCl}$ , filter.

Precipitate=Silica.

---

To filtrate from silica add  $\text{NH}_4\text{OH}$  filter, dissolve precipitate in dilute  $\text{HCl}$ , divide in two parts; in one determine.

Iron and alumina by precipitation with  $\text{NH}_4\text{OH}$ ; in the other determine.

Iron by volumetric method.

---

To filtrate from iron and alumina add  $\text{NH}_4\text{OH}$  +  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  (amm. oxal.) filter, ignite precipitate and weigh as  $\text{CaO}$  or  $\text{CaCO}_3$ .

Calculate as Lime.

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To filtrate from calcium add  $\text{NH}_4\text{Cl}$  +  $\text{NH}_4\text{OH}$  +  $\text{NH}_4\text{NaHPO}_4$ . Allow to stand 12 hours, filter, ignite, precipitate and weigh as  $\text{Mg}_2\text{P}_2\text{O}_7$ .

Calculate as Magnesia.

Subtract total percentages from 100 remainder=Alkalies.

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#### SEPARATION INTO KAOLINITE, QUARTZ AND FELDSPATH.

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Take 1 gr. add water and  $\text{H}_2\text{SO}_4$ , heat for 12 hours on water-bath, decant off clear liquid, treat residue twice alternately with dilute  $\text{NaOH}$  and  $\text{HCl}$ , filter, wash and weigh.

Residue= Quartz and Feldspath.

Analyze residue according to previous scheme or as follows:  
Moisten with water, add 1 c. c.  $H_2SO_4$  and 5 c. c. hydrofluoric acid, evaporate, heat on sand-bath, dissolve residue in water, add  $NH_4Cl + NH_4OH$ .

Precipitate=Iron and Alumina. Multiply iron and alumina by 5.41.

Result=Feldspath.

Subtract from feldspath + quartz.

Result=Quartz.

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To complete the analysis, precipitate the calcium with  $(NH_4)_2C_2O_4$ , filter, ignite.

Weigh as Calcium Oxide or Lime.

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Evaporate filtrate from calcium to dryness, ignite, dissolve in small quantity of water, and  $NH_4OH + (NH_4)_2CO_3$ , filter, dry, precipitate, ignite and

Weigh as  $MgO$  or  $MgCO_3$ .

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Determine alkalies in filtrate by evaporation with  $H_2PtCl_6$ .



## CHAPTER VI.

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As stated in a previous chapter, one of the most important parts of a clay analysis is the mechanical division into various degrees of fineness. Research on this subject has been carried far enough to enable us not only to determine the proportion of certain sizes of grains in the clay, but also to approximately decide to what class these grains belong, whether clay, quartz or undecomposed rock. The mechanical analysis goes even further than this, and shows the state of division of the quartz and feldspath. In the manufacture of the finer clay-wares this is very important, and if carried out with a reliable apparatus, and if the data from which the results are calculated are of the right kind, in some cases, more may be learned from this analysis than from the chemical, but still a large amount of work will have to be done by the expert chemists, before the results of the mechanical analysis can be generally used.

Such properties of the clay as leanness or fatness, porosity, shrinkage, absorption and retention of water, etc., etc., which are very difficult to estimate from the chemical analysis, can be predicted with some certainty from the mechanical.

There are practically two principles made use of in this form of investigation, namely, sifting and sedimentation (or settling in water). The former determines merely the size of the grains, while the latter also takes into consideration such things as the specific gravity, shape, etc., therefore it is far more valuable, not only because it can be carried a great deal further, but also because it takes into consideration more of the qualities which influence the behavior of the burned product. The sifting process is limited, of course, by the mechanical difficulties of making a small meshed sieve.

The principle of sedimentation has been utilized in several methods and styles of apparatus for the analysis of clays, but it must be admitted with regret, the simpler the apparatus the less use it is, and the more complicated and difficult to use, the more valuable. About the most simple form yet pro-

duced is the graduated cylinder. The clay has first to be separated into its natural sized grains by boiling with water for a long time, and is then put into a cylindrical glass vessel which is divided into equal lengths. If the mixture of solid and liquid is left undisturbed for some time, the heaviest and largest grains will settle first, and the lightest and smallest last. The method of determining the relative amounts of these is purely comparative, and consists in noting the time when the top of the layer of sand reaches each mark and the time when the water finally clears. The times noted for the layer to reach each mark are then compared with other determinations, and the results utilized in that way. The method is very faulty, however, and should never be adopted because of its simplicity, as the possibilities of error are so great that the results are never reliable, and an unreliable analysis may do more harm than none at all.

The apparatus generally adopted among ceramic chemists is that devised by Schoene for the analysis of soils. The principle was used before in several ways, but Schoene perfected the apparatus and did away with the chief sources of error, so that the results, if properly obtained and used, are extremely reliable. If a grain of sand is placed in perfectly still water, it will fall to the bottom at a speed varying according to its weight, shape, etc. If the water is made to ascend in a stream of exactly the same speed as that at which the grain of sand fell, the latter will remain suspended in the water; but as soon as the speed of the ascending stream of water is increased beyond that point, the grain of sand will be carried upwards and away.

Let us suppose that the clay to be analyzed is placed in a suitable vessel after being thoroughly disintegrated, and a stream of water sent upwards through the vessel. At the very lowest velocity or speed, only the finest particles of clay will be washed out, and as the velocity of the stream is increased, the particles that are washed out become larger and larger, until at last a sufficient speed has been attained by the water to carry off the largest grains of sand.

In the natural conditions of the clay there will be no sharp divisions between the different sizes, on the contrary, they will vary by imperceptible degrees from the finest to the coarsest, so it has been found necessary to fix certain arbitrary limits. The way this was done was to take a very slow stream of

water, collect all the grains washed off by this and measure a large number of them with the help of the microscope. In this way the size of the largest grains that could be washed off by that velocity was found. Then the velocity of the stream was increased to another point, the grains collected and measured, and thus the largest particles that could be washed away by that velocity determined. This has been done for about five sizes of grains, and most mechanical analyses are made in conformance with these determinations, as it is not necessary for every analyst to measure the grains himself, but merely to measure the velocity of the stream of water.

After the separation into the five sizes of particles, chemical analysis has been employed to determine what these are composed of. The principal work in this line has been done by Seger, whose measurements of the grains and stream velocities are generally considered standard. This form of mechanical analysis is called elutriating or washing out with water, and is also known as slumming.

Before describing the apparatus, and how to make it and set it up, it is well to say that only a very skilled mechanic can make the glass parts properly, and unless they are properly made they are quite useless. The whole apparatus can be imported from Germany, but that is quite expensive, because of the duty, so it is being made for individual use in this country. It was employed in its present form by Schoene for the investigation of soils for agricultural purposes and then utilized by Seger for clays. For most men unaccustomed to such work, the putting together and arranging the apparatus described would be very difficult, even after the glass parts have been properly and accurately made. The work of figuring out the various velocities and constants is difficult for any one who is not a mathematician. It would save much time, and in the end money, if the apparatus could be obtained ready for use, with the velocities all figured out. The description of how these are obtained should be read, however, so that an intelligent idea may be had of how the process is carried out.

Its construction is as follows:—

Fig. 18 shows the vessel in which the actual elutriation or slumming takes place. A stream of water enters at D, and after passing round the bend C, discharges directly into the middle of the conical portion and thence into the cylinder A B.

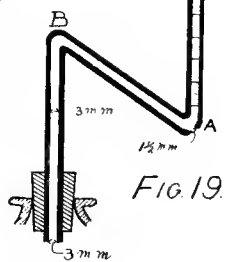
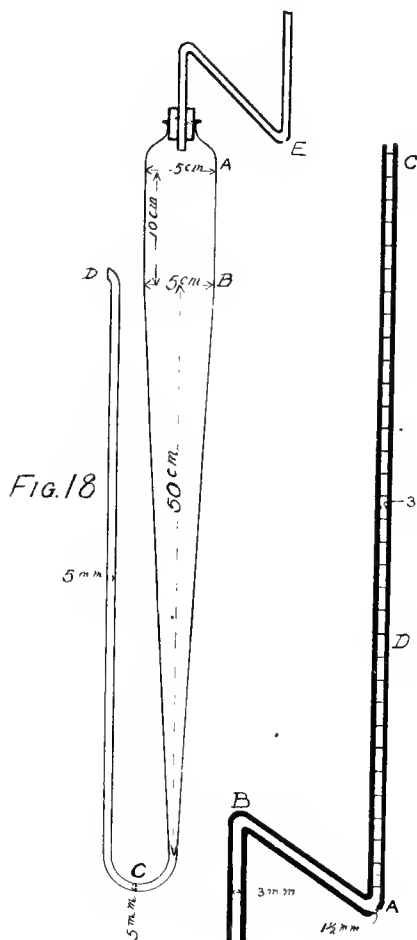
It is in this cylindrical chamber that the water does its work. According to the velocity of the stream, all grains of a certain size are carried upwards and out of the delivery tube at E. This tube is arranged to act in the capacity of pressure gauge as well as delivery tube, so that the velocity of the chamber A B, may be read off on the upright tube A C (Fig. 19).

There are certain points which have to be observed in making this part of the apparatus, and unless they are observed the great advantage of this form over all others is thrown away. The main object is to have a stream of water working on the suspended particles of clay, in such a manner that under the same conditions it is always the same, and always does exactly the same thing. Also some arrangement is necessary by which the velocity can easily be read off.

In order that the stream of water may behave always alike, the flow must be uninterrupted and quite central, so that the direction is straight forward and there are no disturbing side currents. This is ensured by having the bend at C (Fig. 18) an exact semicircle, and the internal diameter of the tube D C exactly the same all through. This should be 5 millimeters or 4 m. m., but never less than 4 m. m., 5 m. m. is preferable. Whatever it is, it must be the same the whole length of the tube and especially so in the bend C. The conical portion B C (Fig. 18) must be perfectly central and true, so that the stream of water is able to spread out gradually and evenly, and reaches the lower part of cylindrical chamber in an even steady stream. This may be tested by blowing strongly in at D, and if the stream of air passes out of the center of the neck it is all right; if it strikes more on one side than the other, then some portion of the apparatus is out of plumb.

The length from B to C should be 50 c.m. and the internal diameter at B should be 5 centimeters. The part A B must be perfectly cylindrical and upright, and should have an internal diameter of about 5 c.m., and a length of 10 c.m. It is very important that this should be a perfect cylinder, but it is not absolutely essential that these measurements be adhered to, between 4 and 5 c.m. for the diameter, and 9 and 11 c.m. for the height are near enough. The neck should be large enough to take a rubber stopper, as shown in the illustration.

Fig. 19 shows the combination of outflow tube and pressure gauge, which is made of carefully selected barometer tube,



the internal diameter of which is exactly the same in every part. This internal diameter should be as nearly as possible 3 millimeters. The external diameter is not important, it may be 7 or 8 m.m. The tube should be bent into a Z-shape at about the angles shown in the figure. The bends at A and B must be very carefully made so that the internal diameter is in no case decreased or increased. This is very difficult but absolutely necessary. At A there should be a circular opening 15 m.m. diameter, with the edges melted quite smooth. The upright portion C. A. should be a little over 1 meter in length and should be divided off into centimeters of which the lowest ten should be divided into millimeters. This may be done by etching with hydrofluoric acid or by pasting a paper scale at the back.

After the glass parts are all satisfactorily obtained a water tank is necessary. This is shown in Fig. 20, A. D. C. It should be made of metal, preferably zinc, to hold about two gallons of water. It must be perfectly airtight with an opening (B) on top for pouring in the water, which can be closed tightly with a screw cap or large rubber stopper. C is an upright piece of glass tube bent at the lower end so that it will fit in a rubber stopper let into the side of the tank. This is for the purpose of supplying air to replace the water that runs out and works in such a way that the pressure of the water in the tank is neutralized by the resistance offered to the air in entering, so that the pressure at the stopcock F is always the same whether the tank is half or quite full.

The top of the tube C should be a little above the top of the tank and the lower end which passes through the rubber stopper should be exactly on a level with the discharge pipe D, which should be as near the bottom of the tank as possible. This outlet pipe D should be connected with a stopcock F, either by a rubber or glass tube, and the stopcock in turn is also connected with the end of the elutriating vessel by a rubber hose. The glass parts should be fixed to an iron support in such a way that both the pressure gauge and the conical vessel are perfectly vertical. This should be ascertained by testing with a plumb line. The top of the pressure gauge may be fastened with a clip fixed to the shelf at E. To receive the discharged water and clay the beaker H is placed on a ring attached to the stand G.

When everything is satisfactorily arranged several prelim-



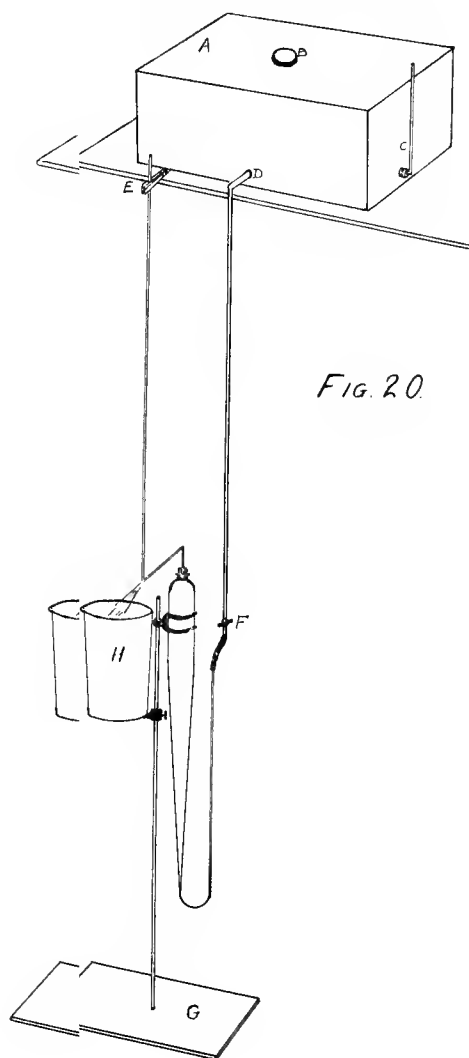


Fig. 20.

inary determinations have to be made before the apparatus can be used. The first thing to do is to find the exact diameter of the cylindrical part of the vessel. The rubber stopper with the pressure gauge should first be taken away and then water run from the tank until it is just above the top of the conical portion B in Fig. 18. The level of the water should be marked by sticking on a label and left for some time to see that it remains in the same position, in case the stopcock leaks. After making sure that this is all right, a measured quantity of water is run from a burette through the neck of the vessel, and the rise in the top of the water measured; a cubic centimetre at a time is enough to add. These additions and measurements should be continued until the sides of the vessel begin to curve inwards and the cylindrical form ceases, which is shown by the addition of one c.c. of water occasioning a greater rise than formerly. When this point is reached it should be marked and the distance between the highest and the lowest part of the cylinder accurately measured and expressed in centimetres.

Let us call this height  $h$ . The addition of each c.c. of water should have caused a rise of exactly the same amount; if there is any large deviation the apparatus should be rejected or if it is near either the top or the bottom all above or below that part should be left out of our calculation. Having found out what portion of the cylinder is perfectly true its total capacity should be found by adding together the separate amounts that were poured into it and calling this  $X$  cubic centimetres. We can then easily find the diameter. We know that the volume of a cylinder is equal to the height, times half the diameter squared, times 3.1416 (or 22).

$$X = h \cdot \frac{22}{7} \cdot \left(\frac{D}{2}\right)^2 \text{ and } D = \sqrt{\frac{14 X}{11 h}}$$

Therefore if we multiply  $x$ , the capacity expressed in cubic centimetres, by 14 and divide that by 11 times the height expressed in centimetres, and then take the square root of the quotient this will be the diameter expressed in centimetres. The next thing to be determined is the velocity in the cylindrical chamber corresponding to the height of the water in the pressure gauge.

If instead of having the upright tube A C, Fig. 19, we have a

simple bent tube for discharging the water, we should have to measure the amount of water that flowed out in a certain time whenever we altered the stopcock; in fact it would be impossible to do more than guess at the velocity in the cylinder. But if the opening at A is small and the graduated tube is placed as shown in the figure, the water begins to rise in the tube, when the flow of water is so fast that it cannot escape from the opening at A. After rising to a certain height it remains stationary and by noting the height and also finding the rate at which the water runs out of the opening we find the index for a certain velocity in the cylinder. This however, is complicated by certain factors, chief of which is the capillary attraction of the narrow tube, which attraction raises the water just as much when it stands at 1 m.m. as when it stands at 1 metre. Schoene determined the value of these influences in the following manner: If it were not for this capillary attraction which we will call C, the following relation would hold good  $(Q_1)^2 : (Q_2)^2 :: h_1 : h_2$  where  $Q_1$  and  $Q_2$  are the numbers of c.c. of water flowing out in one second at the pressures of  $h_1$  and  $h_2$  respectively. But taking into consideration the capillary attraction constant C the following proportion holds good:

$$(Q_1)^2 : (Q_2)^2 :: h_1 - C : h_2 - C.$$

If we work out this equation with several values for Q and h, and find C in each case we can take the average of these and use this all through. To work out the value of C we use the following formula:

$$\sqrt{h_1 - C} = \sqrt{h_2 - C} :: Q_1 : Q_2$$

$$C = \frac{(Q_1)^2 h_2 - (Q_2)^2 h_1}{(Q_1)^2 - (Q_2)^2}$$

If we set the stopcock so that the pressure gauge reads 1.6 c.m. and find that 24.36 c.c of water flows out in one minute (60 seconds)

$h_1$  would be 1.6 and  $Q_1$  would be  $\frac{24.36}{60} = .406$  or the quantity flow-

ing out in a second. In the same way we set the stopcock again so that the gauge reads 60 c.m. and 288 c.c. flows out in one minute, then  $h_2 = 60$  and  $Q_2 = 4.8$  then

$$C = \frac{(.406)^2 \times 60 - (4.8)^2 \times 1.6}{(.406)^2 - (4.8)^2} = 1.18 \text{ c.m.}$$

We do this for several pairs of observations, always a high one with a low one, and find the average for C. This average we adopt for our calculation.

To find the velocity in the cylindrical chamber we proceed as follows: The velocity means the number of millimetres of a certain column of water that will pass one point in a second.

$$V = Q \times \frac{14}{11D^2} \text{ cubic centimetres.}$$

Q is the amount flowing out in one second, D is the diameter of the cylinder, and V is the velocity. We find this for any value of Q, noting at the same time what h is. For instance, if h is 1.2 and Q is 0.406, then V would work out to 0.50 m.m. with a cylinder diameter of 3.227 m.m. Having obtained V in one case we find all other values of V by the following formula, instead of measuring the outflow Q of each value h

$$V \cdot V_n = \sqrt{h - C} : \sqrt{h_n - C}$$

$$V_n = \sqrt{h_n - C} \times \frac{V}{\sqrt{h - C}}$$

$$\text{and } h_n = (V_n)^2 \times \frac{h - C}{V^2} + C$$

where  $V_n$  is the the velocity for any height  $h_n$ . We found C to be 1.18 c.m. and V we just found to be 0.50 m.m. then if we want to know the velocity at a pressure of 30 c.m. if

$$N = \sqrt{30 - 1.8} \times \sqrt{\frac{5}{1.6 - 1.8}} = 4.06 \text{ m.m.}$$

This means that if the gauge indicates a height of 30 c. m. the column of water in the cylinder is passing any one point at the rate of 4.06 m.m. per second. The value  $h_n$  can be

worked out in exactly the same way. When this has been done for all the velocities which we are likely to use, the apparatus is ready for use. Of course, this must be done for each separate vessel and none of the figures just given can be actually used, they are only examples.

The velocities may also be worked out without finding any of the constants by actually measuring the outflow  $Q$  for about 15 or 20 different heights in the pressure gauge and calculating the velocity in each case. These velocities and corresponding heights are then measured off on a piece of paper the points connected, and a curve drawn through all paper (ruled in squares for plotting curves, called coördinate the points. This curve will enable one to tell the velocity for any height in between the heights we have used. Any one familiar with the use of a curve for such purposes will readily see how it is done and if the operator has not used such a thing before it would be too hard to explain, so he had better ask some mathematician to draw it for him.

Seger gives the following velocities and dimensions of grains and they are the standards usually accepted.

#### CLAY.

All grains with a diameter less than 0.01 m.m. washed out by a stream of 0.18 m.m. velocity per second.

#### SILT.

All grains between 0.01 m.m. and 0.025 m.m. diameter washe dout by a stream of 0.7 m.m. velocity per second.

#### DUST SAND.

All grains between 0.025 m.m. and 0.04 m.m. diameter with a stream of 1.5 m.m. velocity per second.

#### FINE SAND.

All grains between 0.04 m.m. and 0.33 m.m. diameter.

#### COARSE SAND.

All with a diameter above 0.33 m.m.

The coarse sand is taken out by sifting so that after all the dust sand is washed out with a velocity of 1.5 m.m. the fine sand can be washed out with any higher velocity, or it may be found by taking out everything which is left in the vessel after the dust sand is got rid of, and weighing it.

The method of analysis is as follows: We take 5 grains of clay, dried at 100°C, but not ground in a mortar. It is absolutely essential that the clay should not be broken with anything harder than the fingers. This clay is placed in a beaker with about 150 c.c. of distilled water, a few drops of hydrochloric acid added, boiled for some time, then allowed to cool and settle. When quite clear the supernatant liquid should be poured off without disturbing the sediment, another 150 c.c. of distilled water poured in, boiled for an hour, and then poured into a sieve with a mesh of as nearly 0.33 m.m. as can be obtained. The sand should be washed through with as little water as possible, aided by rubbing the finger gently over the gauze and everything that goes through caught in a large beaker. What remains on the sieve should be collected, dried and weighed. The mixture in the beaker that came through the sieve should be allowed to settle, most of the clear liquid poured off, and then the rest of the mixture emptied into the elutriating vessel through a funnel placed in the neck. When everything has been washed into the vessel, the pressure gauge should be replaced and the stopcock turned on, so that a velocity of 0.18 m.m. is obtained. This should be kept up till the water in the upper part of the cylinder is perfectly clear. All the liquid passing over at this velocity should be collected, allowed to settle, the clear water poured off, and the residue transferred to an evaporating dish, dried and weighed. This gives the weight of the finest grains in 5 gr. clay, and when multiplied by 20 gives the percentage. Exactly the same method is followed for the other two velocities 0.7 m.m. and 1.5 m.m. by merely opening the stopcock till the corresponding height is reached, allowing it to run until the water is clear, and then passing on to the next velocity. At last we have left in the elutriating vessel nothing but the fine sand, and instead of washing that out, it is simpler to shut off the water, disconnect the apparatus by taking out the pressure gauge, and pour the contents of the vessel through the neck into a beaker, washing out with a good stream of water. This is allowed to settle, evaporated and weighed as before. The following examples give some idea of how the mechanical analysis shows the properties of the clay:

A, is a fat, plastic, refractory, fire clay.

B, much leaner and shorter.

C, a very good fire clay.

D, a clay with a sharp grained sand, shrinks much and checks strongly.

	A.	B.	C.	D.
Coarse sand .....	0.58%	0.34%	0.18%	21.20%
Fine sand .....	3.00%	28.46%	10.64%	11.58%
Dust sand .....	3.52%	13.84%	9.08%	1.84%
Silt .....	11.16%	20.24%	10.88%	2.14%
Clay .....	82.18%	37.10%	68.32%	62.00%

#### HOW TO USE THE RATIONAL ANALYSIS.

The following are a few examples of rational analyses of clays, which have been tested in a furnace, and they are given so that some idea may be formed from the analysis as to the refractoriness of clay.

A kaolin of the following composition, clay 96.55%, feldspath 1.15%, quartz 2.30% was extremely refractory, and its shape was unaltered at the highest temperature brought to bear on it, namely 2,500°C, the melting point of platinum. At this temperature its surface was slightly glazed, but otherwise it appeared unaltered. The next kaolin containing 74.08% clay, 8.7% feldspath, 17.21% quartz, began to glaze at 1400°C, the melting point of steel, and at 2500°C the cone-shaped lump had bent over and almost lost its shape. The poorest kaolin, whose composition would, but for its color, have classed it among the ordinary clays, began to glaze at 1200°C. and at 2500°C, had melted down to a formless patch like a piece of glass. Its composition was as follows, clay 55%, feldspath, 21.6%, and quartz 23.6%. Finally, a clay of extremely low refractoriness had the following composition:—46.52% clay proper, 6% feldspath, and 47.48% of quartz. This began to be affected by a heat of 1000°C, melting point of silver, at 1400°C it had lost its form, and at 1600°C it was molten and almost boiling.



## ATOMIC WEIGHTS FOR CLAY ANALYSIS.

---

Aluminum.....	Al.....	27
Barium.....	Ba.....	137
Bromine.....	Br.....	80
Calcium.....	Ca.....	40
Carbon.....	C.....	12
Chlorine.....	Cl.....	35.4
Copper.....	Cu.....	63.2
Fluorine.....	F.....	19
Hydrogen.....	H.....	1
Iodine.....	I.....	127
Iron.....	Fe.....	56
Magnesium.....	Mg.....	24
Manganese.....	Mn.....	55
Nitrogen.....	N.....	14
Oxygen.....	O.....	16
Phosphorus.....	P.....	31
Potassium.....	K.....	39
Silicon.....	Si.....	28
Sodium.....	Na.....	23
Sulphur.....	S.....	32
Zinc.....	Zn.....	65



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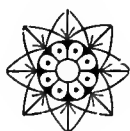
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